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Saturable Absorbing Materials For Q-Switching Neodymium Lasers

FINAL REPORT

By

Hai-men Lo

MAY 1972

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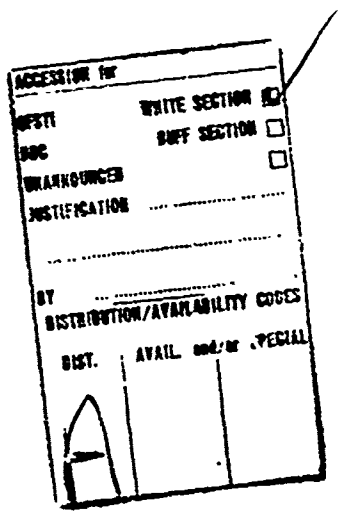

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13. ABSTRACT

This report describes investigative work performed on IBM proprietary dye 106-5 for Q-switching neodymium lasers, carried out under Contract No. DAAB07-71-C-0122. This dye was used in solution form to Q-switch a neodymium glass laser to deliver a peak power of over 100 megawatts in approximately 20 nanoseconds and in plastic thin film form to Q-switch a flashlamp pumped neodymium-yttrium aluminum garnet (Nd:YAG) laser to deliver a peak power of over 3 megawatts with a 15 nanoseconds pulse-width. A plastic thin film with liquid cooling also proved to be useful in Q-switching a multiple pulse Nd:YAG laser. The selection of solvents and solid host materials for this dye, as well as the construction of the liquid-dye cell and the plastic-dye film, are discussed. Results on durability and temperature stability tests for both the dye solution and the plastic-dye film are also included. Based on the present results, design recommendations are made for improving the performance of the Q-switch dye in a solution form, as well as in a solid matrix.

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SATURABLE ABSORBING MATERIALS FOR Q-SWITCHING NEODYMIUM LASERS

FINAL REPORT

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This report describes investigative work performed on IBM proprietary dye 106-5 for Q-switching neodymium lasers carried out under Contract No. DAAB07-71-C-0122. This dye was used in solution form to Q-switch a neodymium glass laser to deliver a peak power of over 100 megawatts in approximately 20 nanoseconds and in plastic thin film form to Q-switch a flashlamp pumped neodymium-yttrium aluminum garnet (Nd:YAG) laser to deliver a peak power of over 3 megawatts with a 15 nanoseconds pulse-width. A plastic thin film with liquid cooling also proved to be useful in Q-switching a multiple pulse Nd:YAG laser. The selection of solvents and solid host materials for this dye, as well as the construction of the liquid-dye cell and the plastic-dye film, are discussed. Results on durability and temperature stability tests for both the dye solution and the plastic-dye film are also included. Based on the present results, design recommendations are made for improving the performance of the Q-switch dye in a solution form, as well as in a solid matrix.

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Section 1. INTRODUCTION

The potential advantages offered by using Saturable Absorber for Q-Switching lasers have been recognized for a number of years. These advantages include simplicity, smaller laser beam divergence, improved laser efficiency, narrower output spectrum*, etc., as compared to mechanical or electro-optic Q-switching devices.

This report describes investigative work performed on IBM proprietary Q-switch dye 166-5 for Q-Switching neodymium lasers by IBM Corporation from February 1, 1971 to January 31, 1972. This work was supported by the United States Army Electronics Command under Contract No. DAAB07-71-C-0122.

The primary result of this program was successfully using this dye in solution form to Q-switch a neodymium glass laser to deliver a peak power of over 100 MW in approximately 20 nanoseconds and in thin plastic film to Q-switch a flashlamp pumped Nd:YAG laser to deliver a peak power of over 3 MW with a 15 nanosecond pulse width. Tests showed that both the dye solution and the plastic-dye mixture functioned well over Mil Spec storage and operational temperature range. A plastic-dye film with liquid cooling also proved to be useful in Q-switching a multiple pulse Nd:YAG laser.

Section 2 of this report gives the Statement of the Problem. Section 3 describes the dye material used in this work. Section 4 discusses the approach to the problem. Section 5 summarizes the experimental results obtained with the laser. Section 6 gives general summary and recommendations for improving the performance of the Q-switch materials.

*B. H. Soffer, Journal of Applied Physics, 35, 2551 (1964)

Section 2. STATEMENT OF THE PROBLEM

The objective of this program was to develop saturable absorber materials for Q-switching neodymium lasers emitting at 1.06 μm. IBM proprietary Q-switch dye 106-5 was used for this study. The program included the test and the improvement of the Q-switch property of this dye with the intent of meeting the following goals:

a. Energy Handling Capability–At least 500 mj per cm^2 in 20 nanoseconds for 1000 shots without damage or deterioration
b. Reliability–Deterioration free operation over long periods of time while exposed to ambient radiation
c. Repeatability–The laser spectral output, energy content, and pulse shape remain relatively constant (comparable to mechanical switches) for at least 1000 shots without readjustment in the dye concentration
d. Temperature Stability–Relatively insensitive to temperature fluctuations within the range from -40 degrees F to 120 degrees F.

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Section 3. PASSIVE Q-SWITCH

It was suggested by Hellwarth* that the laser can be made to emit giant pulses of radiation if the cavity resonance of the laser is controlled. If the laser oscillation is inhibited until after the population inversion in the laser material has reached a maximum condition, and the regeneration of the optical cavity is suddenly switched from a low gain to a high gain cavity, giant pulses of laser light may be obtained.

The methods of inhibiting the laser oscillation may be grouped into three classifications:

a. Mechanically Active-Rotating prisms or reflectors
b. Electrically Active-Kerr or Pockel cells
c. Totally passive, obtaining the regeneration switching through a saturation of the absorption of a lossy medium by the laser once oscillation has begun.

The advantages of passive Q-switch is simplicity; no moving parts or external fields are required and no critical alignment problems exist. In contrast to active devices that require external synchronization between the pumping lamp and the Q-switch, the passive Q-switch technique is self-synchronizing in that the Q-switching action inherently occurs close to the time for maximum gain in the laser crystal. A passive Q-switched laser has smaller beam divergence than a laser Q-switched with a rotating prism or mirror. It is also more efficient than a laser Q-switched with a Kerr cell or Pockel cell because it eliminates the need for an intracavity polarizer and hence the reduction in output power caused by the birefringence effect in the laser rod.

Most saturable absorber materials used in Q-switching neodymium lasers were found to have degradation problems. The transmission of Kodak 9860 Q-switch dye solution, one of the most widely used dyes, has been measured at 1.06 μm in a Beckman DU-2 spectrophotometer and found to be completely bleached by the light source of the equipment in less than 15 minutes. This degradation not only makes a frequent adjustment of the

*R. W. Hellwarth, Advances in Quantum Electronics, edited by R. J. Singer (Columbia University Press, New York, 1961) pp. 334-341.

dye concentration necessary for maintaining a proper Q-switched laser output, but also makes it difficult to obtain repeatable results. It is necessary to protect this Q-switch solution from the pump light or adjust the solution after every laser pulse.

IBM 106-5 dye is the one that does not have this degradation problem. The transmission curves of the solvent and solution are shown in Figures 1 and 2, respectively. Its decadic molar extinction coefficient at 1.06 μm is 2.5×10^4 mol^{-1} cm^{-1}. Preliminary test of this dye for Q-switching a Nd:Glass laser was done by IBM Corporation in Gaithersburg, Maryland, during 1969. This test was for durability and temperature stability.

In the durability test, the laser was formed with a 40 percent reflective output mirror, a Korad 1 cm thick passive Q-switch cell, which incorporated a 100 percent reflective mirror, and a Korad K-1 laser head, which consisted of a Brewster end 1/2-inch diameter by 6-inch long neodymium glass rod pumped with a helix-type xenon flashlamp. The laser rod was maintained at 25 degrees C through a Korad water cooling system. Laser cavity length was about 40 cm. The total electrical input energy was 4000 joules. Typical Q-switched laser output was 1.7 joules in 20 nanoseconds, which corresponds to a peak power of 67 MW/cm^2 or an energy density of over 1.3 $joule/cm^2$, assuming uniform distribution of energy over a 0.5 inch diameter circular spot. The laser energy and pulse shape were monitored at the same time with an EG&G radiometer and an ITT F4000 photodiode with S-1 surface. The experimental arrangement is shown in Figure 3. A typical output pulse is shown in Figure 4.

The test extended over nine days without any adjustment in dye concentration. The cell, which contained approximately 3 ml of the dye solution adjusted to approximately 65 percent absorption at 1.06 μm, was sealed with a rubber stopper and was not shielded from room light in the laboratory. In over 546 shots made during this period, only five of the pictures showed double Q-switched pulses. These five were randomly distributed throughout the test; therefore, we believe these spurious pulses were due to the fluctuation in the flashlamp output. Figure 5 shows the laser output energy distribution obtained during these 546 events.

No degradation of the dye was observed during or after the test. Tests conducted later with the dye solution which had been prepared at the time of the original durability test, found that the solution functioned just as when it was prepared more than one year before. During this time it was stored in a stoppered bottle exposed to the ambient light.

Temperature effect on this dye was checked by installing the Q-switch cell inside a Tenney temperature controlled chamber. Two extra windows were introduced into the laser cavity, as shown in Figure 6, mounted on

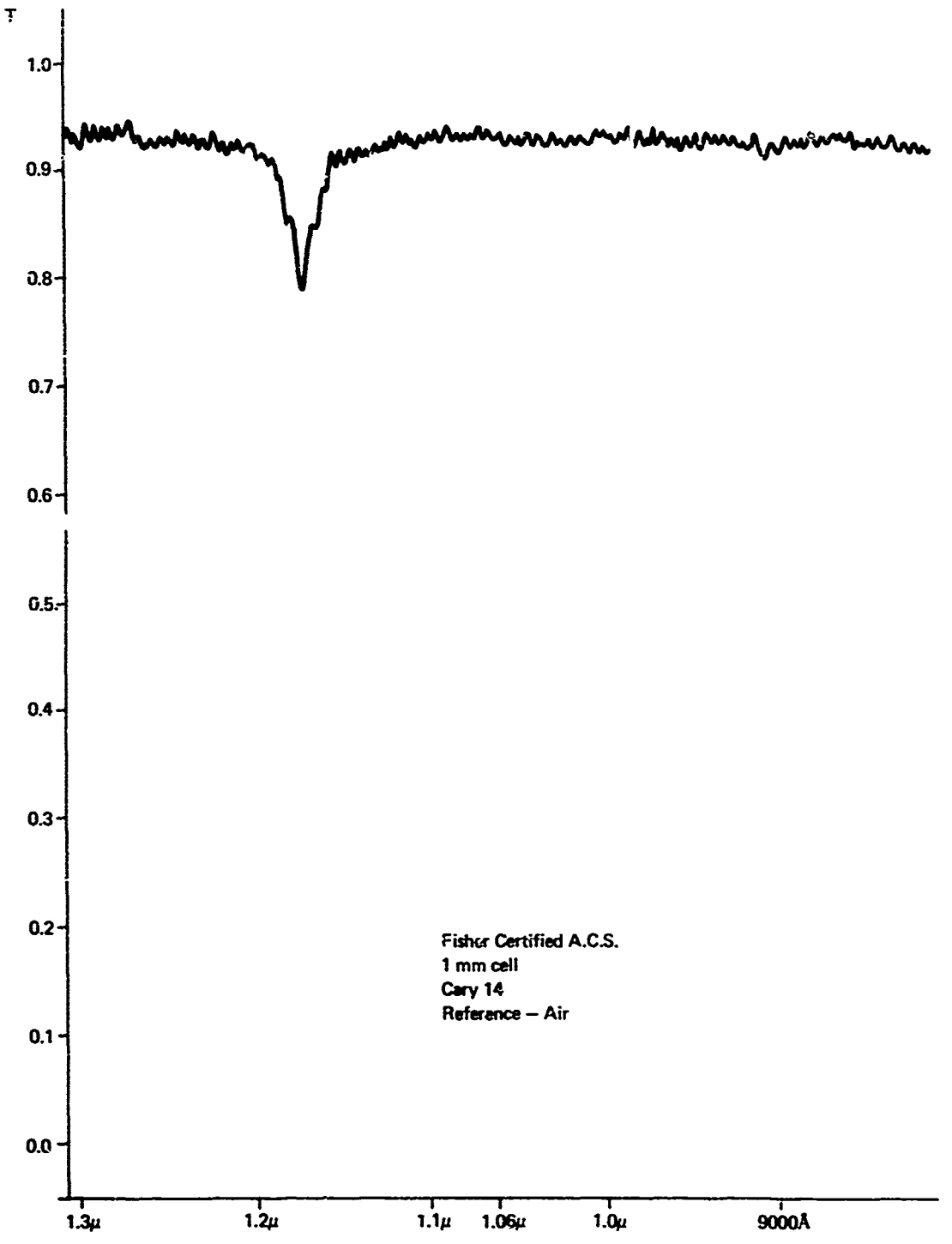

Figure 1. Transmission Spectrum of 1,2-Dichloroethane

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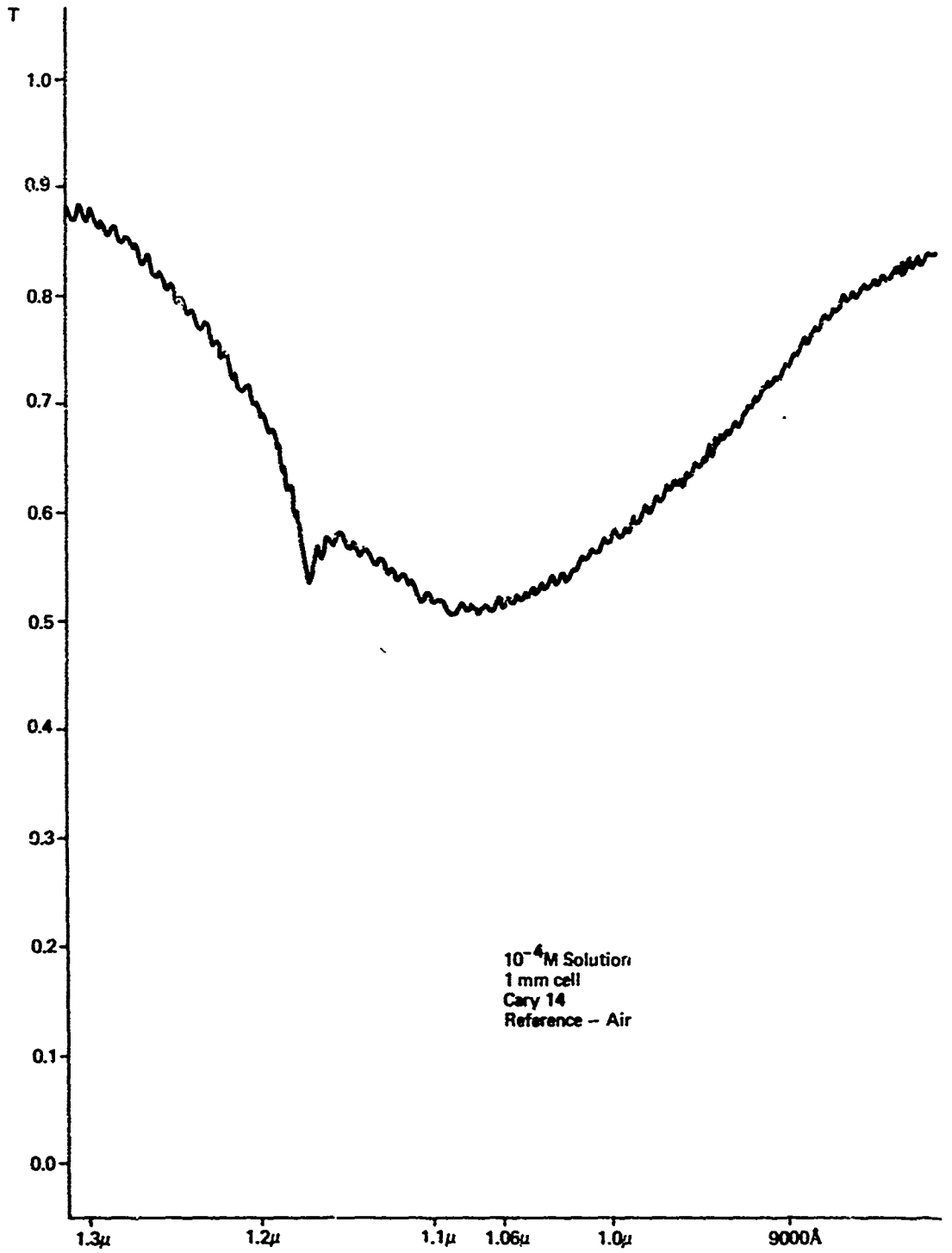


Figure 2. Transmission Spectrum of 106-5 Dye in 1,2-Dichloroethane AB0322-2

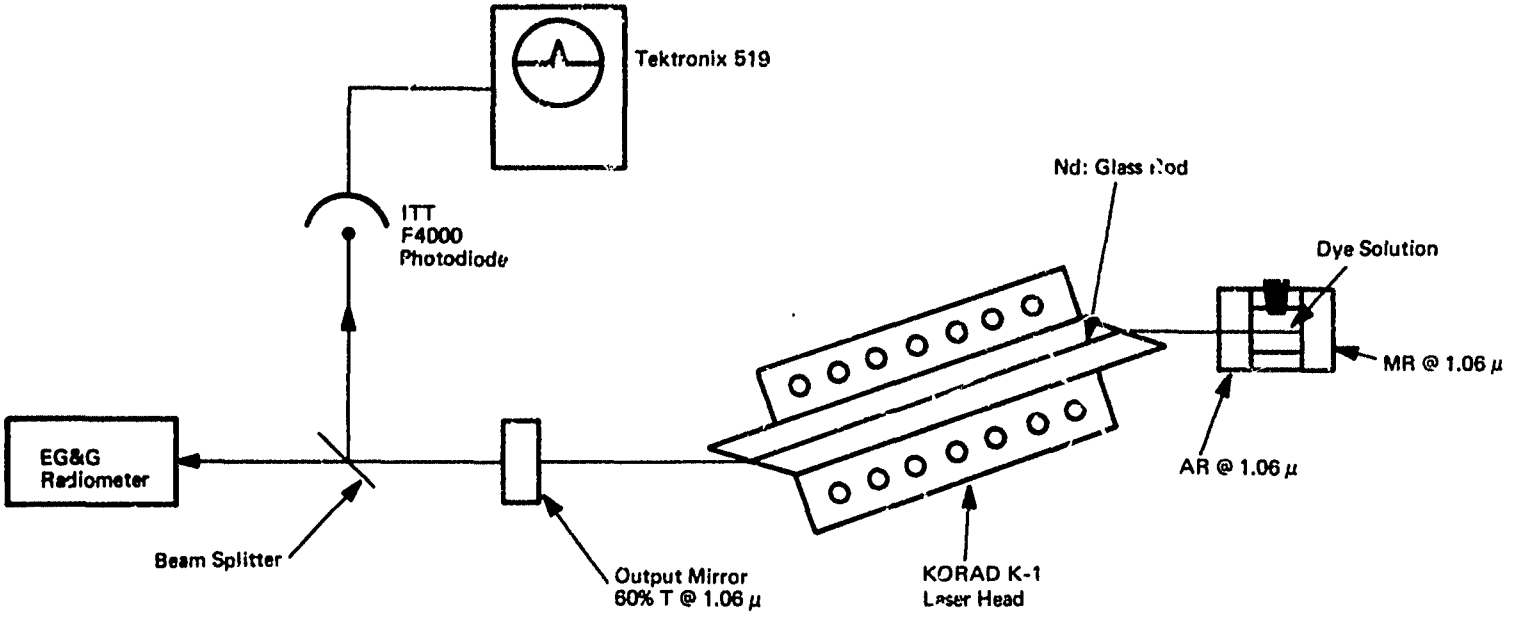
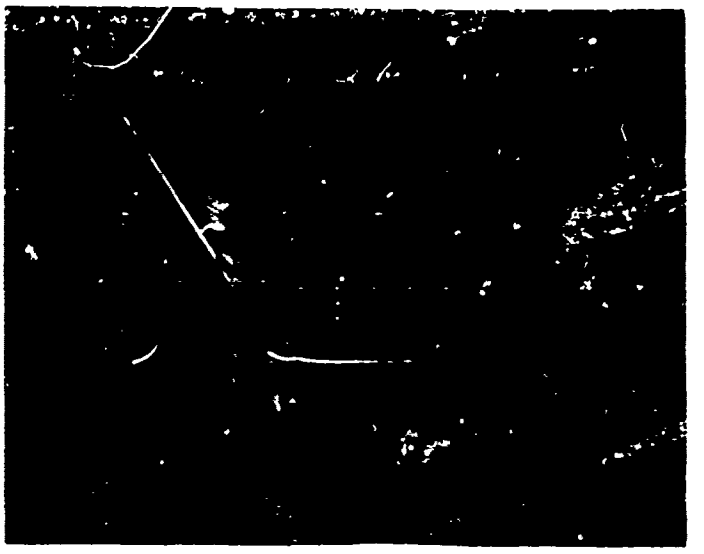


Figure 3. Experimental Arrangement for Testing 106-5 Dye Q-Switch Solution With a Nd:Glass Laser AC0322-2



Passive Q-Switched Nd: Glass Laser Output
Total Energy: 1.98 Joules
Time: 20 ns/div

Figure 4. Passive Q-Switched Nd:Glass Laser Output

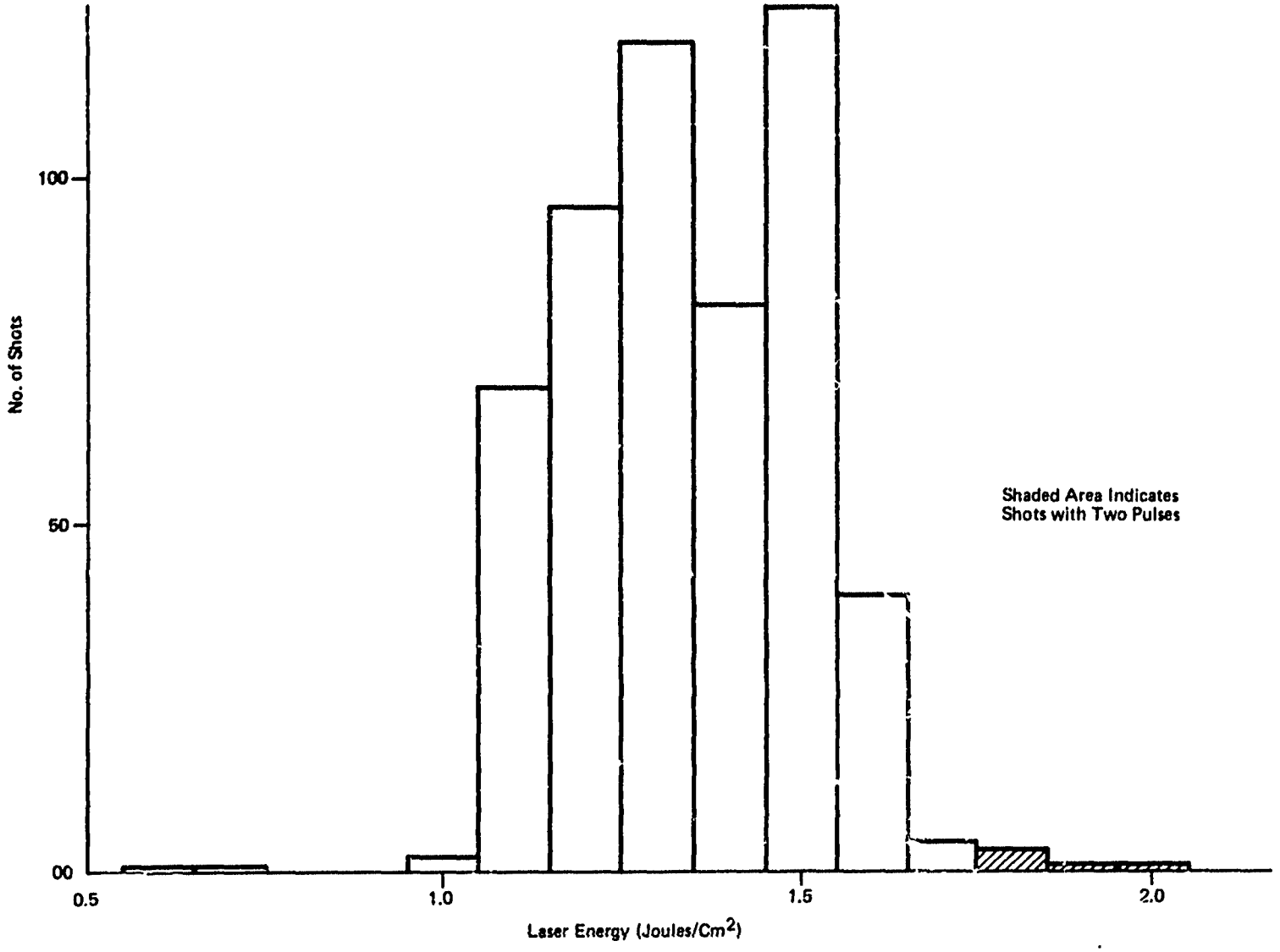


Figure 5. Result of Life Test Using 106-5 Dye Solution (1, 2-Dichloroethane) to Q-Switch a Nd:Glass Laser

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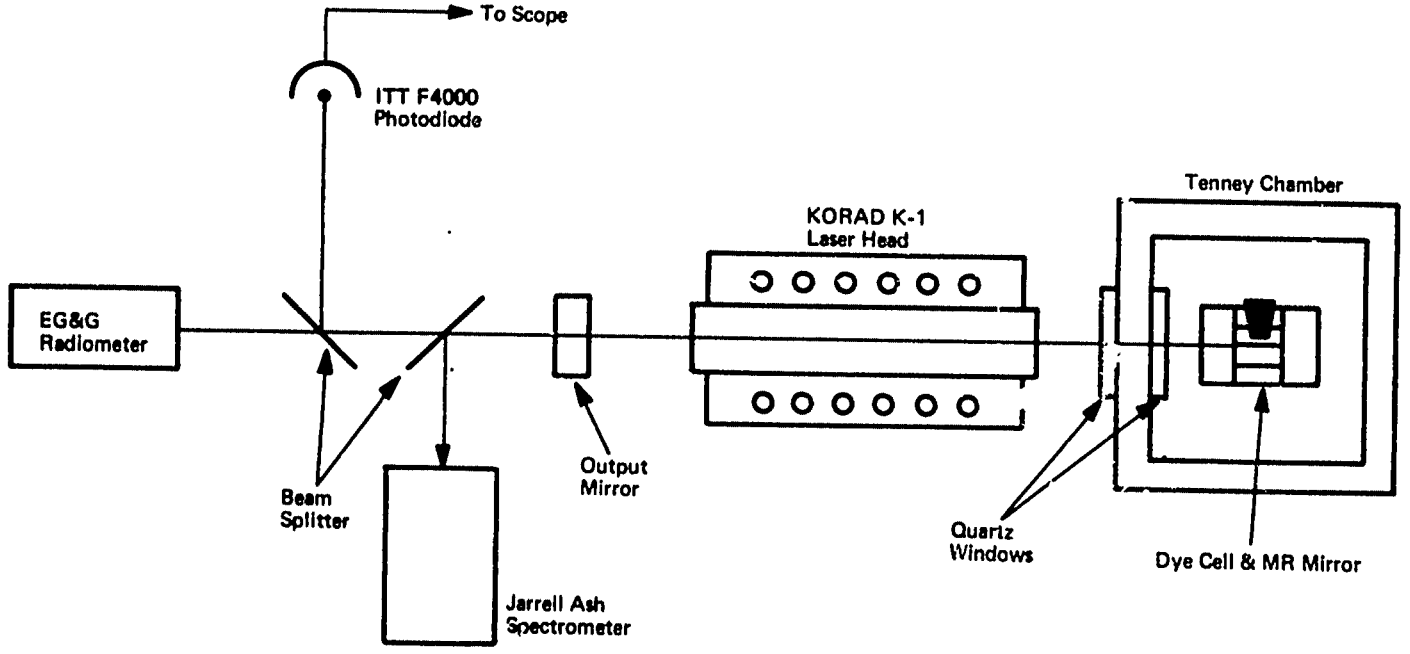

Figure 6. Experimental Setup for Testing Temperature Effects on the Q-Switch Function of the 106-5 Dye Solution

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the inner and outer wall of the entrance hole of the chamber so that not only a constant temperature inside the chamber could be maintained during the test, but also during the low temperature operation no frost would form on the outside surface of the window. The laser cavity in this case was extended to about 50 cm. This test ran from a temperature slightly below the boiling point of the solvent down to its freezing point. Satisfactory Q-switching was found between 70 degrees C (158 degrees F) and -45 degrees C (-50 degrees F). The solution froze at -50 degrees C. No change or degradation was observed in the Q-switch solution over the entire range of temperature. A plot showing the laser output energy versus the temperature of dye solution is given in Figure 7. Wide fluctuation of laser output was mainly due to poor alignment at the dye cell because the inner wall of the Tenney chamber is made of sheet metal which expands irregularly when the temperature is changed.

During the test on temperature effect, the laser spectral output was also checked with a 1-meter Jarrell-Ash Spectrometer at 70 degrees C, 25 degrees C, and -45 degrees C. No significant shift in laser wavelength was observed.

In addition to the above tests, this dye solution was also used for Q-switching a continuously pumped Nd:YAG laser. In this case, provision was made for continuously circulating the dye solution through a 1 mm thick dye cell. The purpose of circulation of this solution was to avoid any loss due to thermal distortion resulting from absorption. Although the average power of the laser output was only 150 milliwatts, the pulse train contained pulses with well regulated spacing and peak power. At fixed pumping levels, one can optimize the peak power of the pulse train by adjusting the concentration of the dye solution. We achieved operation at close to 5 KHz without losing average power. The experimental arrangement and a typical pulse train are shown in Figure 8. This experiment was possible because of the extreme durability of the dye.

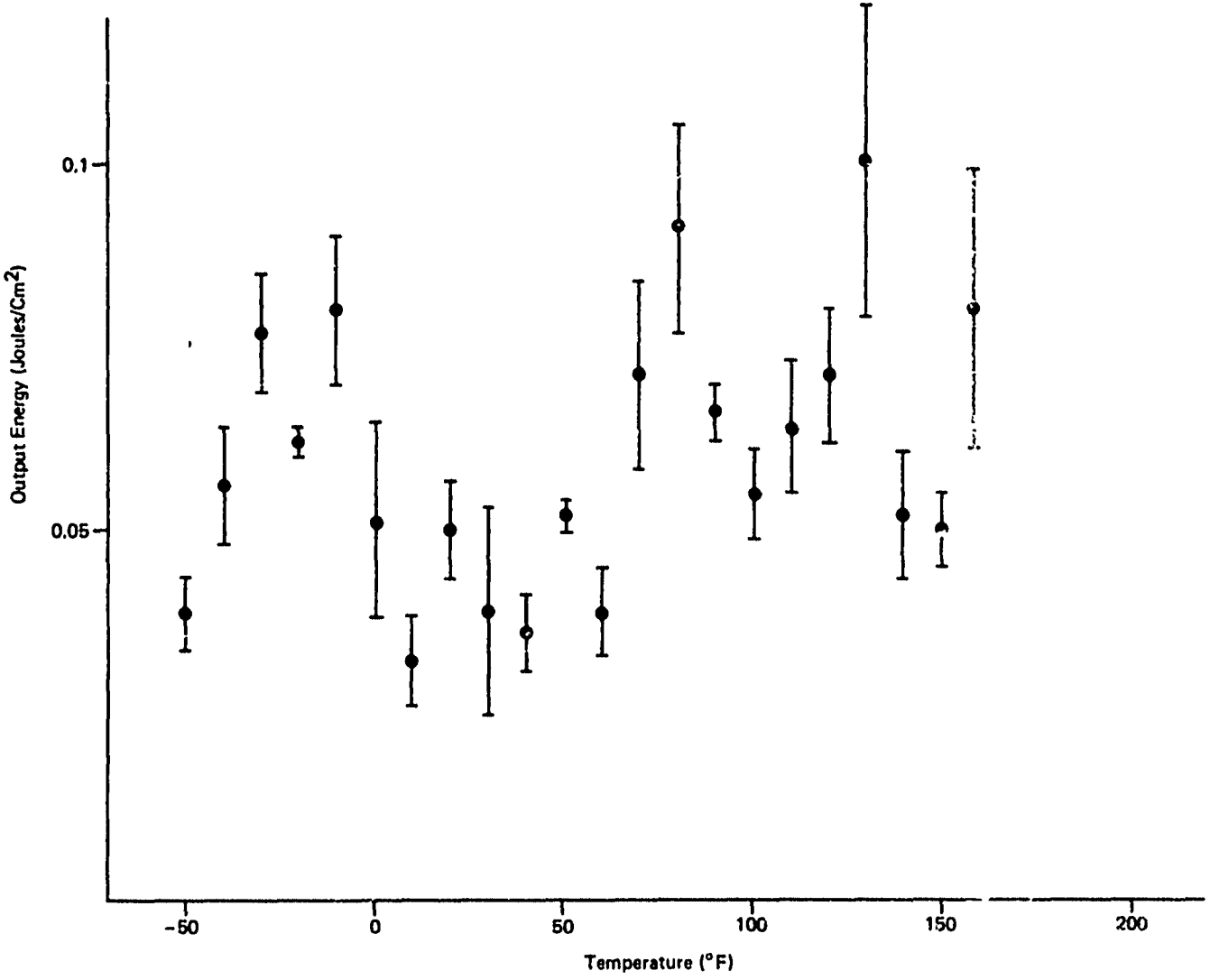

Figure 7. Result of Temperature Test on 106-5 Dye Solution (1, 2-Dichloroethane) in Q-Switching a Nd:Glass Laser

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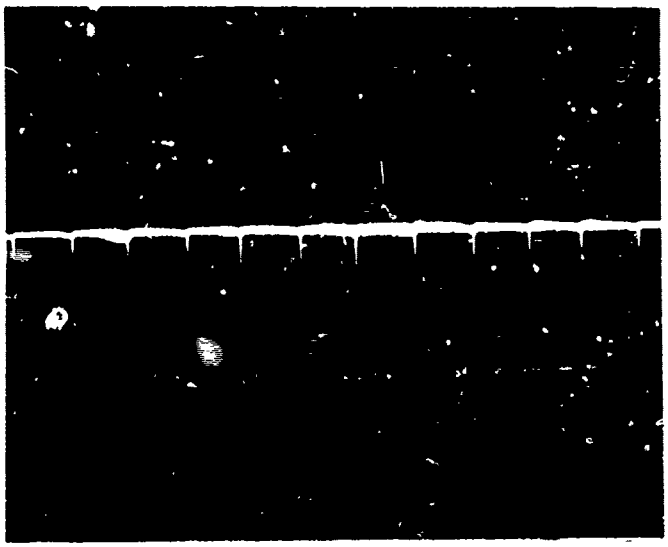
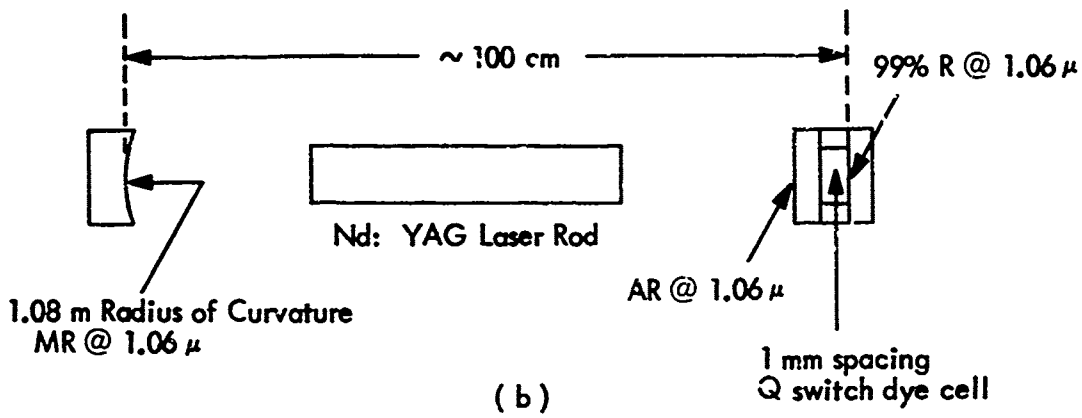


(a)

Time: 200 μs/div; Voltage: 100 mV/div



(b)

(a) CW pumped, passive Q-switched Nd: YAG laser pulse train

(b) Experimental arrangement

Figure 8. Signal and Experimental Arrangement for Using 106-5 Dye Solution to Q-Switch a CW Pumped Nd:YAG Laser

Section 4. SAMPLE PREPARATION

Several valuable experiments which will lead to improved Q-Switch performance were suggested by the previous tests. To achieve better Q-switch performance of the 106-5 dye, the solvent effect was studied. Different solvents shift the peak absorption wavelength of the dye solution and may also effect the relaxation time of the dye material. The durability of the 106-5 dye also suggested that the material might be incorporated in a solid matrix, which could further simplify the Q-switch component.

4.1 SATURABLE ABSORBER IN SOLUTION FORM

106-5 dye functioned well in 1,2-dichloroethane, but this solvent attacked epoxy adhesives used in cells. This necessitated more expensive cell construction techniques than could otherwise be used. Therefore, it was advantageous to find a better solvent and more durable adhesives.

4.1.1 Solvent Selection

In addition to practically no absorption at laser wavelength, a solvent was evaluated on the basis of its useful temperature range, vapor pressure, toxicity, specific heat, density, and viscosity. Table 1 lists the properties of some solvents investigated. Toluene was selected for this project because it has several advantages over other chemicals in the table as solvents for the 106-5 dye. First, the 106-5 dye is more soluable in toluene, which allows a greater range of concentrations to be used. The use of higher concentration would permit use of thinner Q-switch cells, i.e., shorter lasers. Second, toluene freezes at -95 degree C (-139 degrees F) and boils at 110 degrees C (231 degreas F) and will better satisfy the Mil Spec storage and operational temperature requirements. Third, the human toxicity of toluene is lower. The lower specific gravity and viscosity of toluene make flowing easier in case a circulation system is required. The higher heat capacity of toluene makes the thermal distortion resulting from absorption a less serious problem. Additionally, epoxides are much more resistant to toluene.

The transmission spectrum of the toluene used in our experiments is shown in Figure 9. This solvent is spectroquality supplied by Matheson, Coleman, and Bell. Note that there is only a slight absorption in the region around 1.06 μm, the neodynium emission wavelength. A nominal 8 percent loss was incurred in this measurement from reflection at the cell surfaces, since the sample was run against an air reference.

Table 1. Properties of Some Solvents for 106-5 Dye

Solvent	Melting Point	Boiling Point	Specific Gravity	Vapor Pressure	Human Toxicity	Viscosity (in Centipoises)	Specific Heat cal/gm/°C
Toluene $C_6H_5CH_3$	-95°C	110°C	0.866 (d_4^{20})*	40mm (at 32°C)	Cause mild macrocytic anemia	0.55 (at 20°C)	0.40
1,2-Dichloro-ethane $ClCH_2CH_2Cl$	-40°C	83-84°C	1.2569 (d_4^{20})	100mm (at 30°C)	Vapors produce irritation of respiratory tract and conjunctiva, corneal clouding, equilibrium disturbances, narcosis and abdominal cramps	0.80 (at 20°C)	0.30
Chloroform $CHCl_3$	-63.5°C	61-62°C	1.484 (d_{20}^{20})	100mm (at 10.4°C)	Narrow margin of safty as anesthetic, overdoses may cause hypotension, respiratory and myocardial depression, and death	1.20 (at 20°C)	0.23
Methylene chloride CH_2Cl_2	-97°C	40-41°C	1.3255 (d_4^{20})	400mm (at 24°C)	Relatively high, may cause cardiac arrhythmias nausea and vomitting	0.449 (at 15°C)	0.29
Carbon Tetrachloride CCl_4	-23°C	76.7°C	1.589 (d_{25}^{25})	100mm (at 24°C)	Poisoning by inhalation, skin absorption or ingestion of excessive quantities	0.96 (at 20°C)	0.20

*Specific gravity at 20°C referred to water at 4°C

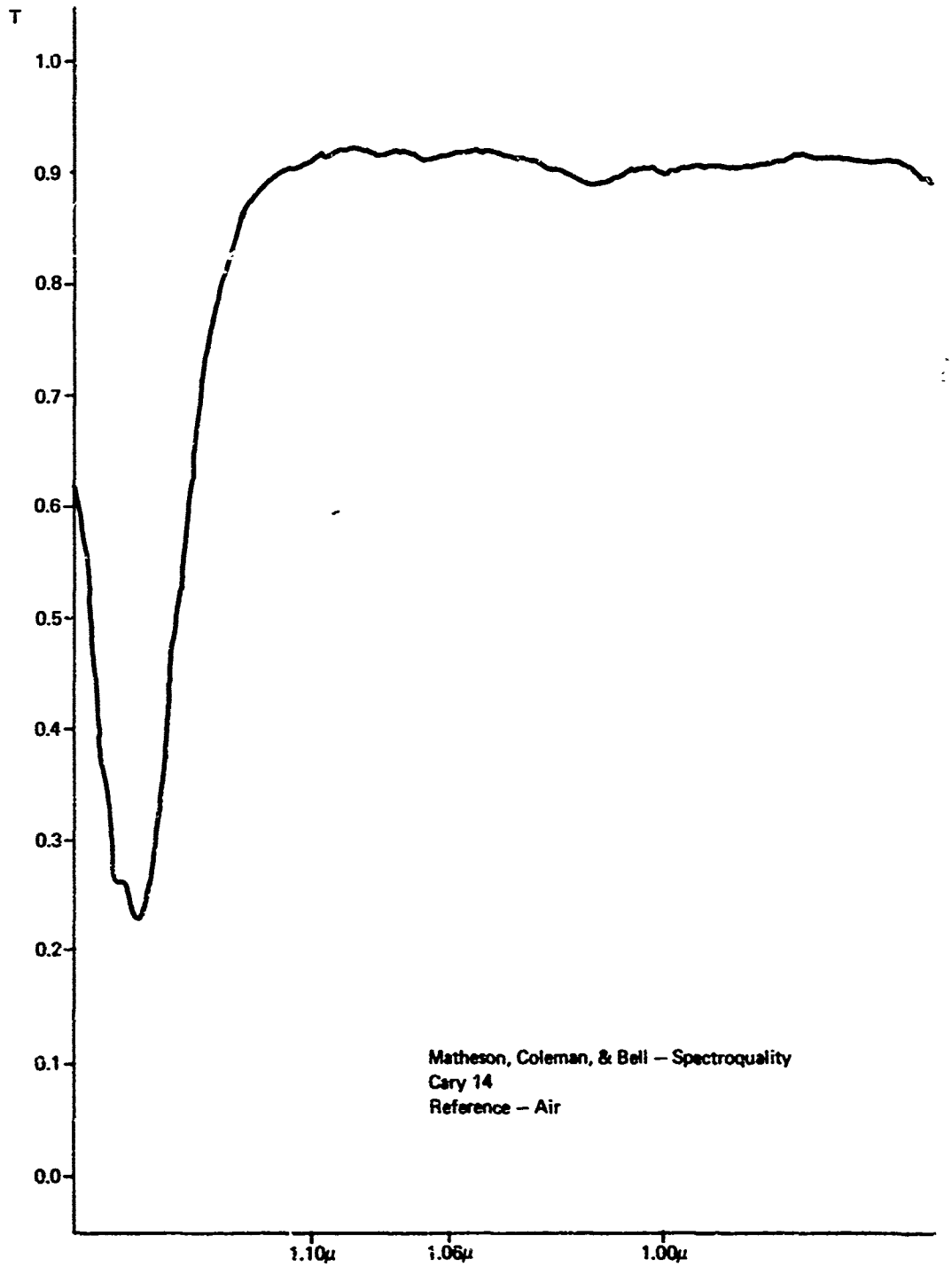


Figure 9. Transmission Spectrum of Toluene

AG0322-2

The transmission spectrum of the 106-5 dye in toluene is shown in Figure 10. Note that the peak absorption was shifted to shorter wavelengths (higher energy) than in the 1,2-dichloroethane. This shift away from 1.06 μm was not great enough to significantly change the absorption at the laser wavelength. Therefore, the compound should still function well as a neodymium laser Q-switch.

Samples of 10^{-4} M solutions of 106-5 dye were prepared in toluene and stored for periodic sampling as a test of stability. One sample was placed in a clean 100ml volumetric flask and the other in a dark glass flask of identical size. Both flasks were exposed to ambient light.

4.1.2 Epoxy Selection

Several epoxy adhesives were tested in different solvents for long last bond. Test samples were made by using each epoxy adhesive to join two 3/8 inch x 1 inch glass plates together and cured at room temperature for over 72 hours, so that maximum bond strength was developed. Individual samples were then dipped into test tubes, which contained different solvents. Evaluation of the epoxy was made by recording the length of time that glass plates held together while immersed in the solvent. Tabulated results are shown in Table 2.

Table 2. Test Results for Epoxy Adhesives in Different Solvents

Solvent / Lifetime / Epoxy Type $ Vendor	Toluene	1, 2-dichloroethane	Methylene Chloride
ECCOBOND 45 Emerson & Cumming, Inc.	< 24 hrs.	< 3 hrs.	< 2 hrs.
ECCOBOND 55 Emerson & Cumming, Inc.	< 5 mos.	< 3 hrs.	< 2 hrs.
ECCOBOND 285 Emerson & Cumming, Inc.	< 2 mos.	< 24 hrs.	< 2 hrs.
HYSOL 931 DEXTER Corp.	> 5 mos.	< 120 hrs.	< 24 hrs.
HYSOL 956 DEXTER Corp.	> 5 mos.	< 120 hrs.	< 2 hrs.

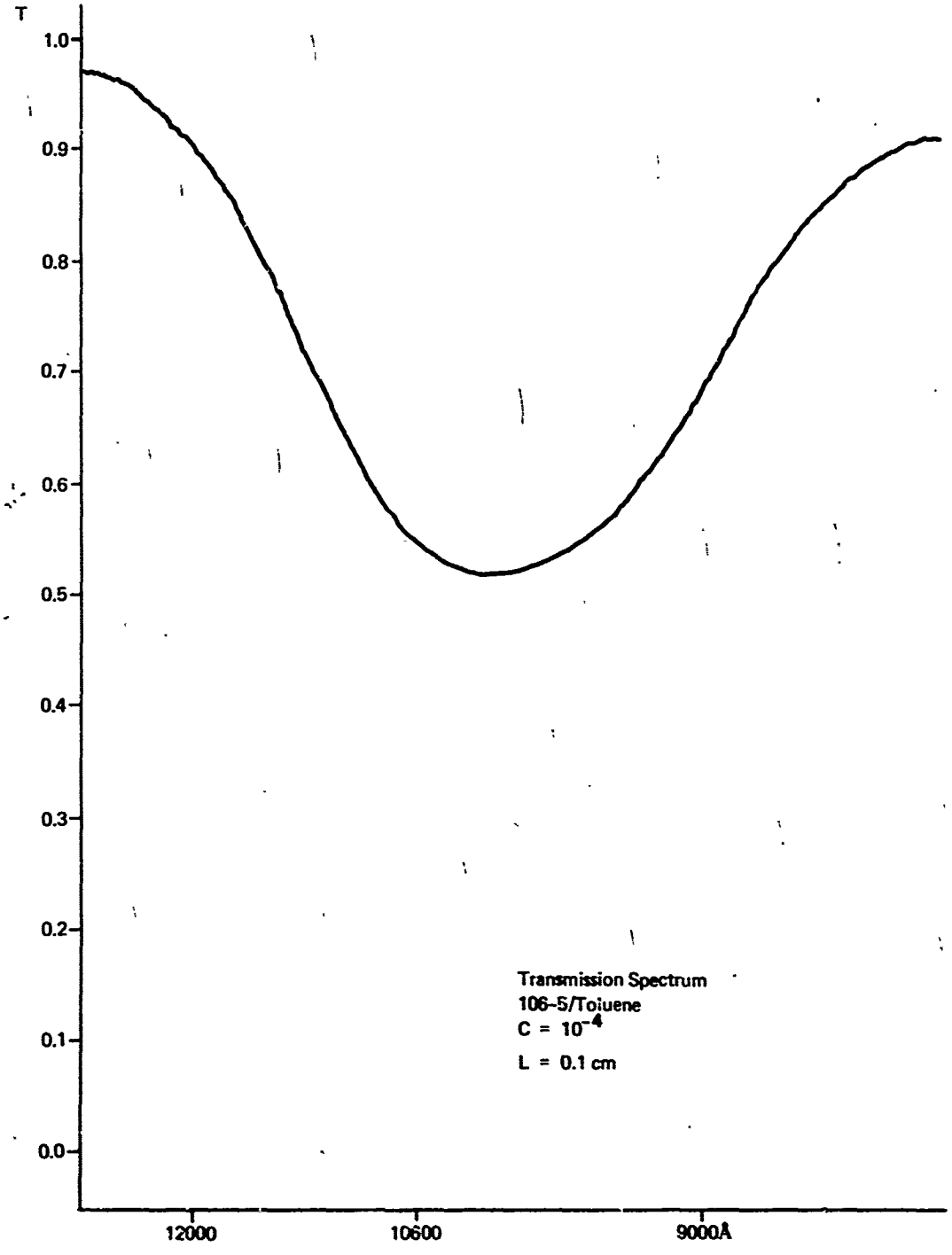


Figure 10. Transmission Spectrum of 106-5 Dye in Toluene

AH0322-2

Test on epoxy adhesives were also done on Hysol 917, 931, and 956 by D. Johnson of IBM-Research Laboratory in San Jose, California. Two types of specimens were used. In one, overlapping glass slides were glued together. In the other, beads of adhesive were placed on a glass slide, so that beads were both immersed and in the vapors above the solvent level. All glass slides were first cleaned with acetone. Samples were cured for 16 hours at 80 degrees C in an oven. No attempt was made to determine optimum cure conditions. The results can be summarized as follows:

Best adhesive: 931 > 956 > 917
Best solvent (attacks least):
toluene > dichloroethane > chloroform > methylene chloride.

4.2 SATURABLE ABSORBER IN SOLID FORM

4.2.1 Selection of Polymers

Several polymers were investigated as host materials for the 106-5 dye. A polymer was selected under the criteria that it has no absorption at the laser wavelength and that the 106-5 dye can be mixed into the polymer uniformly.

There are at least two general approaches to the task of incorporating 106-5 dye in a solid polymer to produce optical quality material. One is polymerization with the dye present and the other is dispersion of the dye in previously polymerized material. Each of these methods has been tried and only the second has been satisfactory. Free radical intermediates, created when polymerizing with the dye present reacted with the 106-5 dye compound, changed its nature. Therefore, polymers were first evaluated by dissolving a small quantity in a proper solvent and casting thin films with the solution in a covered evaporation dish. The results of these experiments are summarized in Table 3. Polystyrene and polymethylmethacrylate (PMMA) were then chosen for further study.

The optical density curves for a piece of undoped polystyrene and undoped PMMA are shown in Figures 11 and 12, respectively. Dye solution was mixed into the polymer solutions for making the plastic-dye films. The 106-5 dye absorption was not significantly altered in either of the plastic hosts from that in 1, 2-dichloroethane solution. This absorption, as represented by the optical density of samples measured with a Cary 14, spectrophotometer, are shown in Figures 13 through 16. Figure 13 shows the absorption peak for 106-5 dye in polystyrene is at 1.06 μm, as desired. The optical density of 0.3, corresponding to 50 percent absorption at low signal levels, is closed to the density used to Q-switch

Table 3. Summary of Polymers

Polymer	Vendor & ID	Relative Molecular Weight*	Solvent	Result
Polystyrene	Dow, Clear Styron Lot 608084 666 U-26-71	89,000	Toluene	Forms good films
Polycarbonate	GE, Lexan 141	100,000	CH_2Cl_2	Film formed, opaque
Polymethylmethacrylate (atactic)	Rohm & Haas Lot 67689 Product 3-0112	141,000	CH_2Cl_2	Forms good films, can be air dried or in oven 60°C
Syndiotatic PMMA	IBM	160,000	CH_2Cl_2	Forms good films
Polychlorostyrene	Cellomer Associates, Inc.		$CHCl_3$	Film formed, uneven quality
n-Butyl/isobutyl Methacrylate	Cellomer Associates, Inc. 50/50 Copolymer		$CHCl_3$	Forms good films, soft

*Measured by Gel Permeation Chromatography

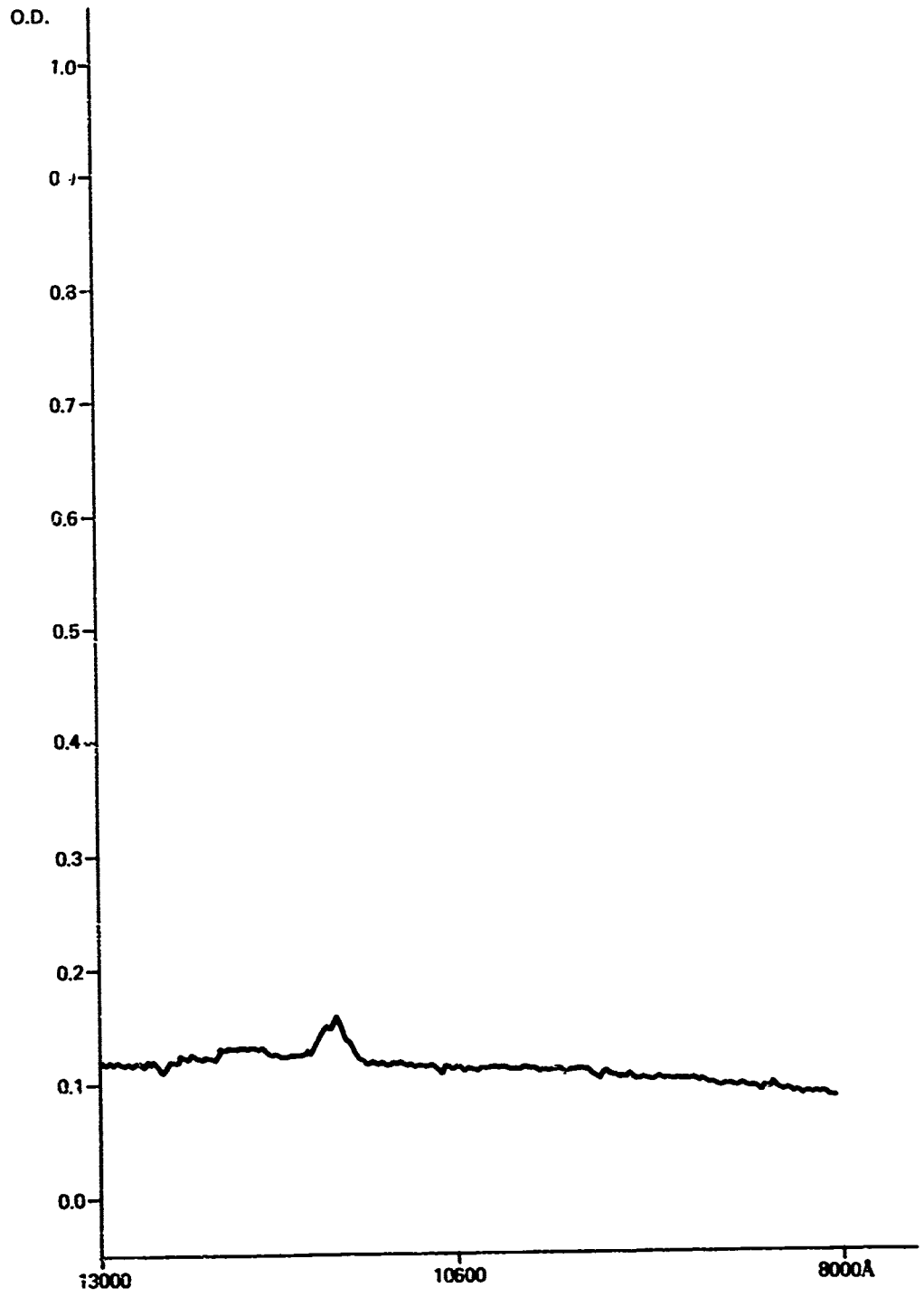


Figure 11. Optical Density of Polystyrene

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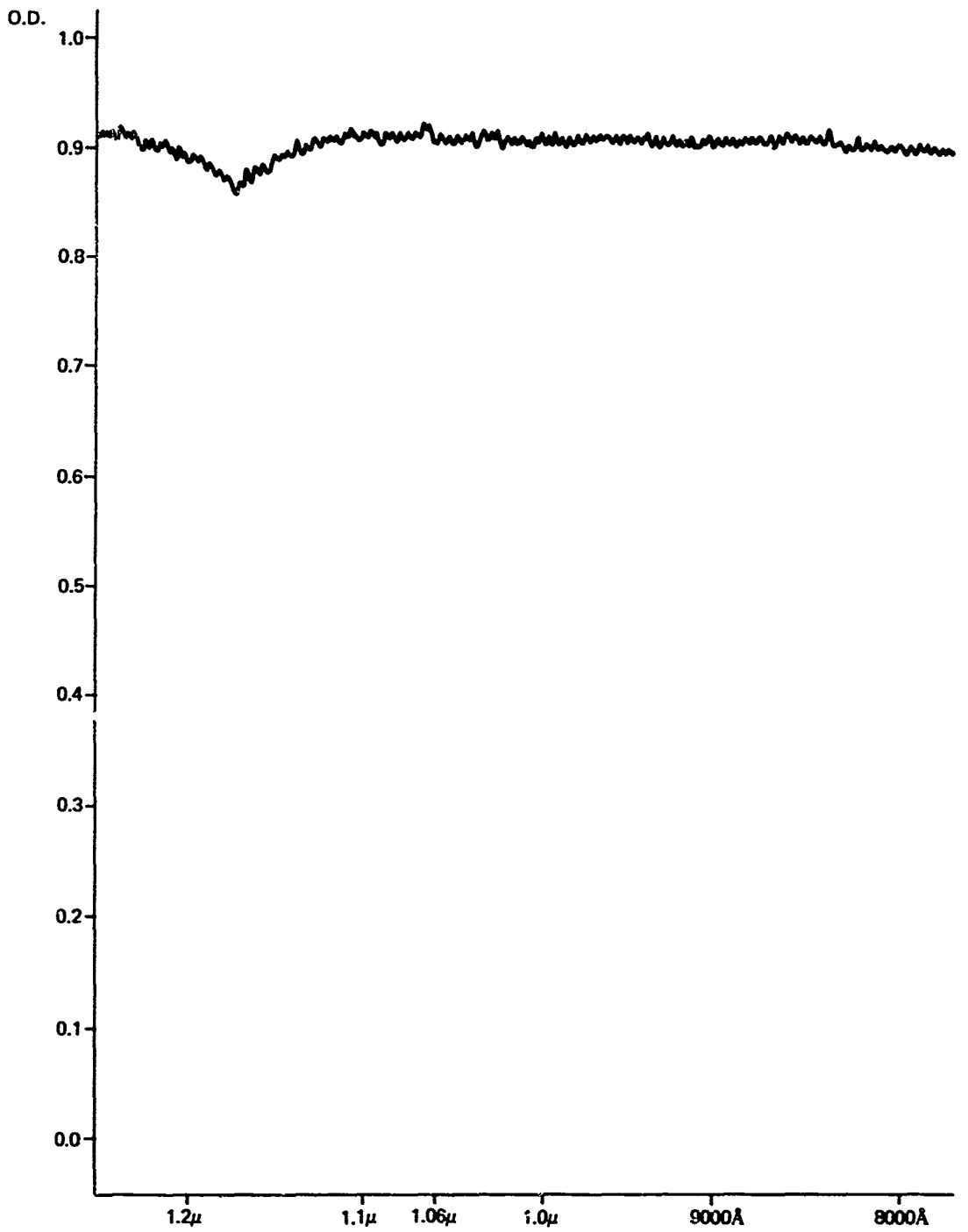

Figure 12. Optical Density of PMMA

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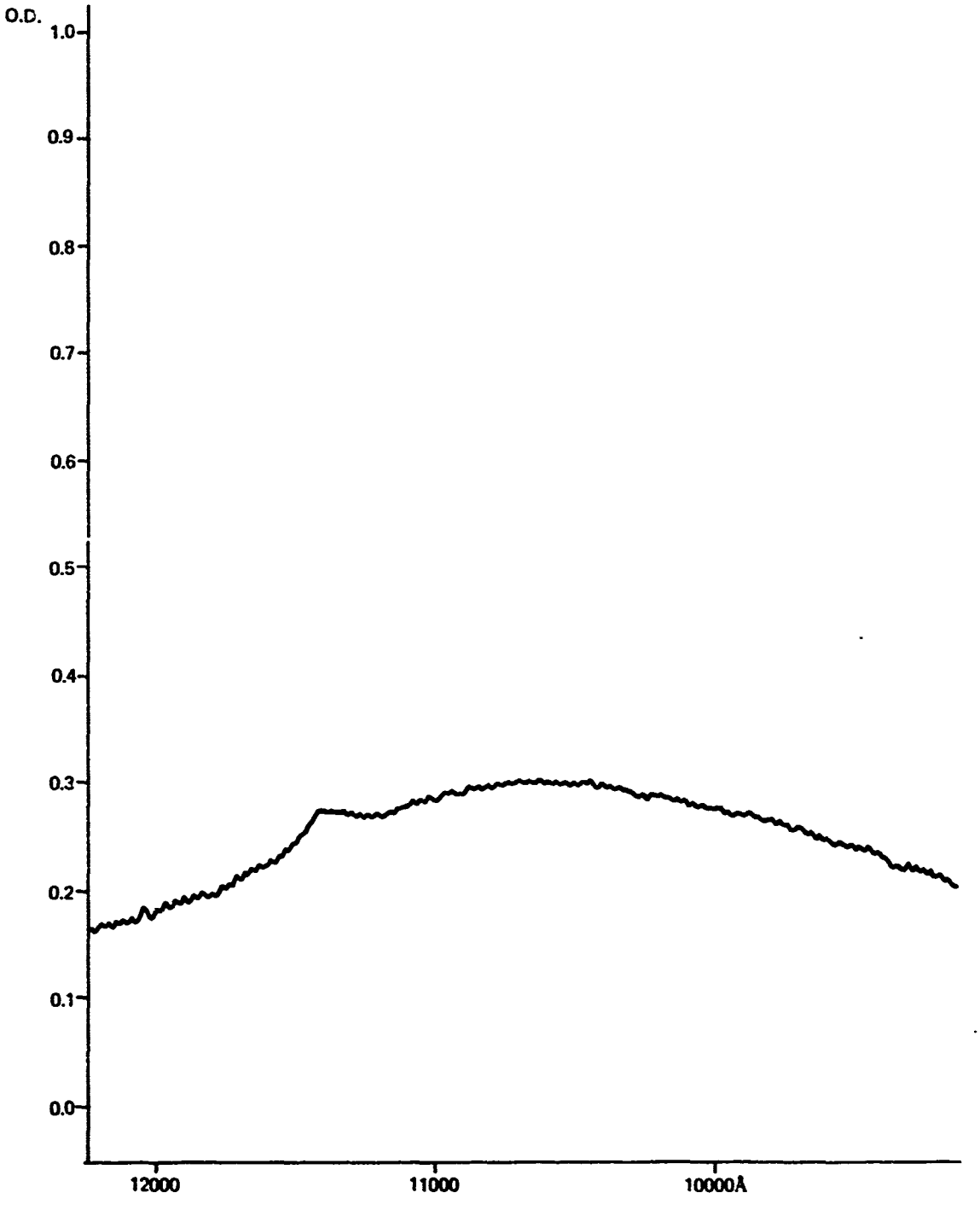

Figure 13. Optical Density of 106-5 Dye in Polystyrene AK0322-2

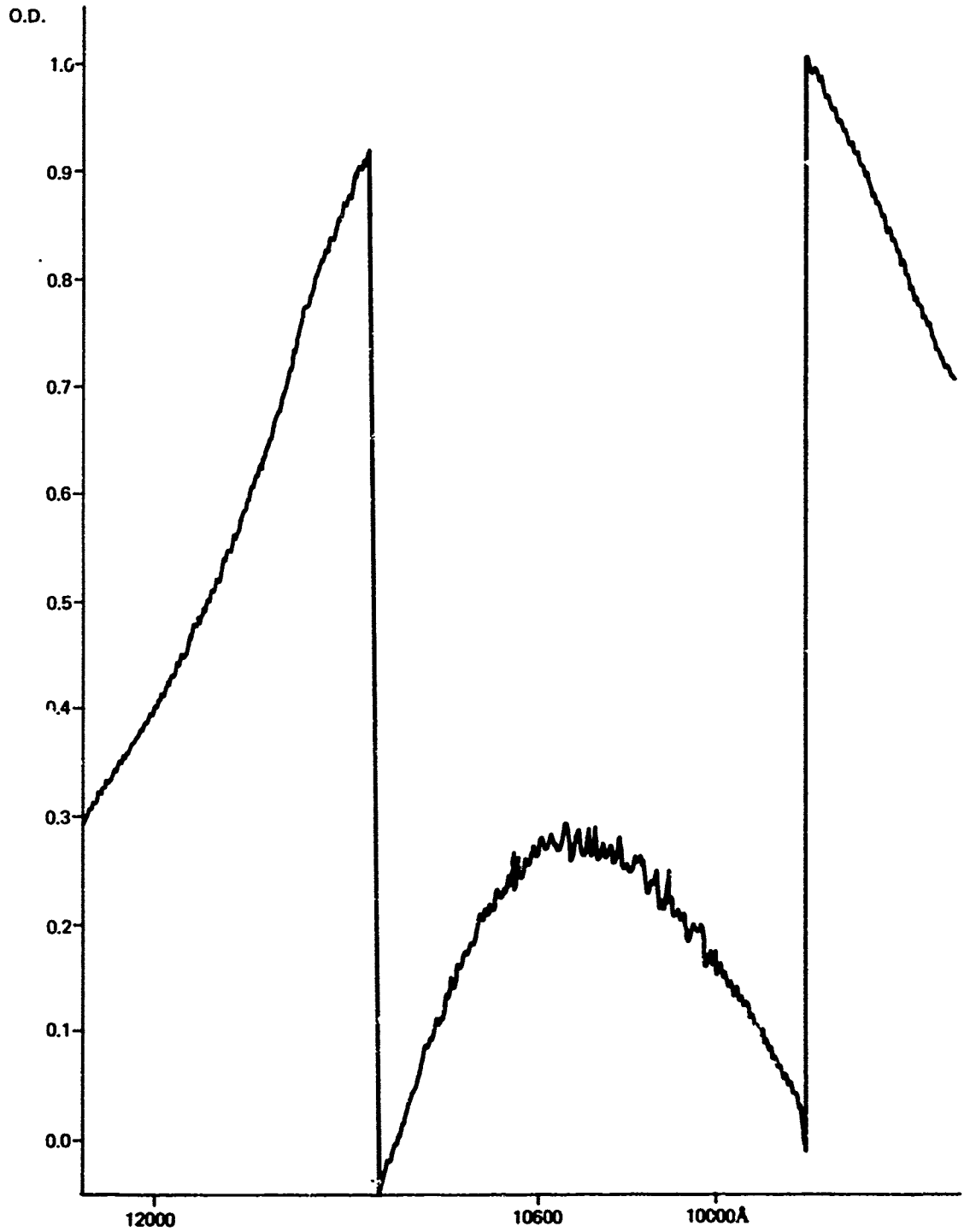

Figure 14. Optical Density of 106-5 Dye in Polystyrene

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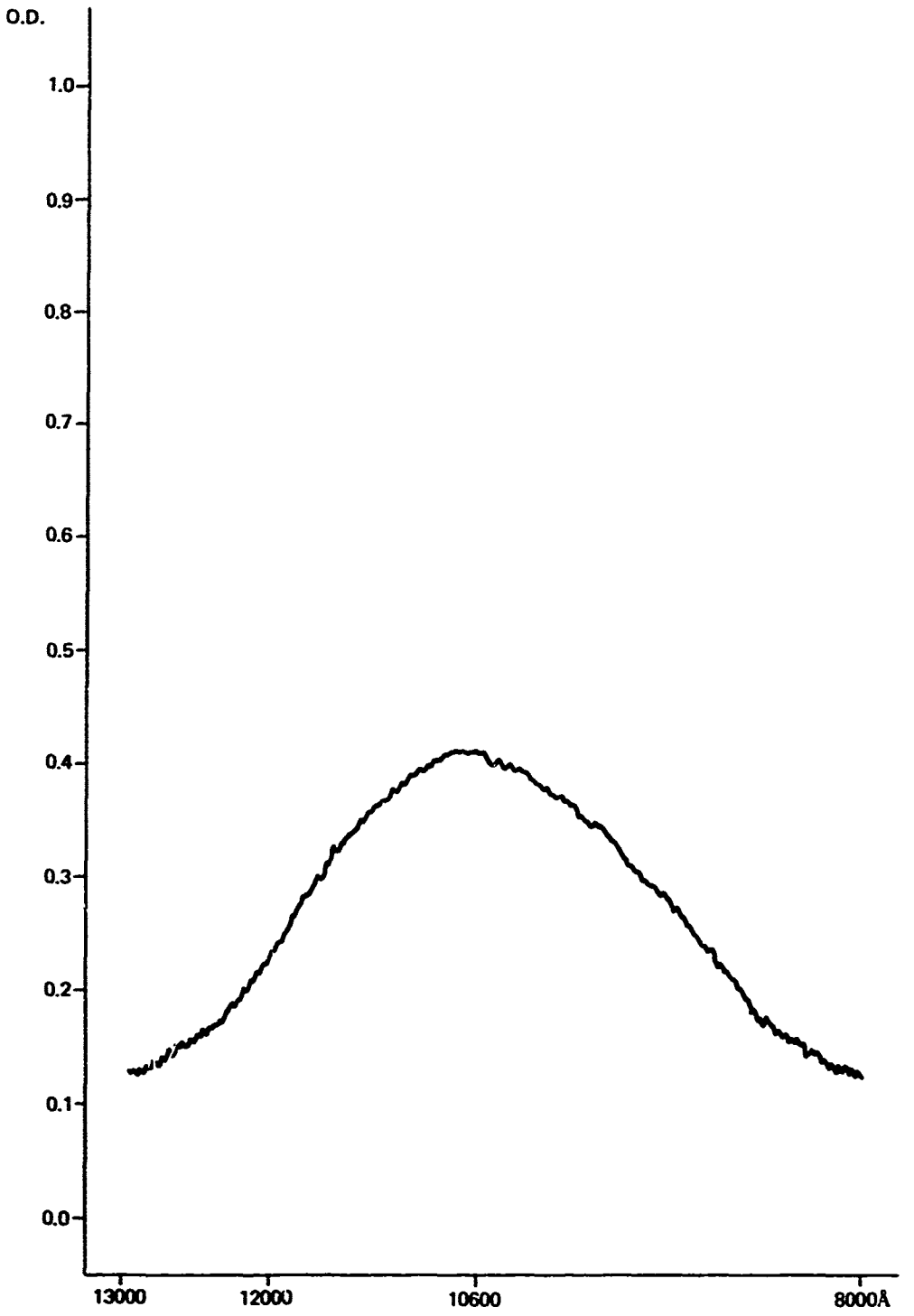

Figure 15. Optical Density of 106-5 Dye in PMMA

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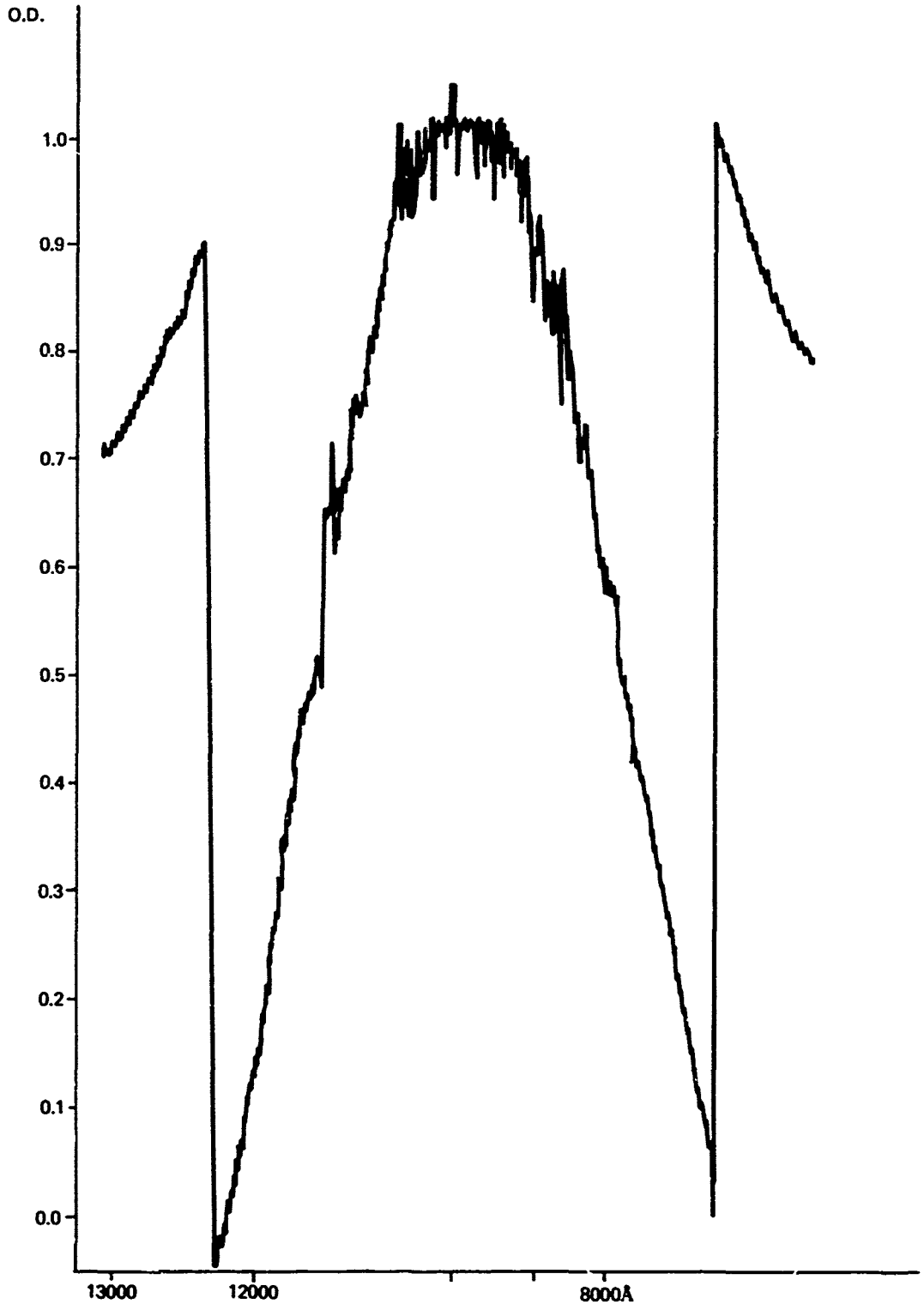

Figure 16. Optical Density of 106-5 Dye in PMMA

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the neodymium glass laser. The small secondary feature at 1.14 μm was due to the absorption of polystyrene. This assertion is verified by Figure 11, which shows a scan of the same spectral region with a sample of undoped polystyrene.

Figure 14 shows the absorption in a more heavily doped sample (O.D.=1.287), using the polystyrene host. The absorption peak was still at 1.06 μm, and no dimer features were observed. This sample was approximately 0.2 cm thick. Optical damage occurred in this sample during measurement of the absorption spectrum. The damage appeared as a contraction and wrinkling in the portion of the sample exposed to the IR spectral source. The absorbed power density was a few watts/cm^2.

Figure 15 represents a sample of 106-5 compound in PMMA host. The peak absorption is again at 1.06 μm.

A PMMA sample of optical density greater than 2 was prepared as shown in Figure 16. No shift of the peak from 1.06 μm was observed. Additionally, neither this sample, nor any other using the PMMA host material showed damage during the IR spectrometer scans.

4.2.2 Casting of Plastic Thin Films

Brief tests were made of the durability in laser beams of unsupported and supported samples of PMMA for finding better ways to adopt the plastic-dye film inside the laser cavity. Undoped PMMA, in thickness up to 0.2 cm, was not damaged by the focused output of a 3 watts CW Nd:YAG laser. It is believed that this result would also obtain thicker samples. Unsupported (cast without backing substrate), doped samples were damaged by this CW laser beam, which was focused to an intensity of 30 kW/cm^2. This film (∿0.01 cm) with low doping density (a few percent absorption) damaged readily when supported on a glass substrate. A similar film cast on a copper substrate was not damaged. This result indicated that further tests should be made on metal mirrors suitable for Q-switched laser use.

The initial technique on casting thin film for use inside a laser cavity was tried on a 1" diameter maximum reflection mirror with dielectric coating. The solution was made by dissolving 4 grams of PMMA (Rohm & Haas, Optical Grade) in 100ml of methylene chloride (CH_2Cl_2). To this solution, sufficient Q-switch compound was added, until the concentration was about 4×10^{-5}M. This dye was previously dissolved at higher concentration in toluene, and the solutions were then mixed. A predetermined amount of the PMMA-dye solution was then placed in a receptacle formed by a hollow teflon cylinder, closed at the bottom by the mirror surface

on which the film was supported. This solution was then allowed to evaporate slowly to dryness in a dust free environment.

Two films of about 0.1 mm thickness with a transmission of 74 percent at 1.06 μm were cast by evaporating 1.5 ml of the PMMA-dye solution on a 1 inch diameter maximum reflection coated dielectric mirror. The plastic film did not adhere to the dielectric coating well. Both surfaces of the film appeared smooth. There was no control on obtaining uniform film thickness over entire surface.

Films coated on a 1 inch diameter gold coated stainless steel mirror also did not adhere to the mirror surface well.

To obtain uniform film thickness and to have the film supported on both surfaces, one chooses to cast it between two parallel windows. The first attempt was to make a thin cell with two pieces of microscope slides, using a sheet of 5 mil thick H-film cut into U shape spacers as shown in Figure 17. These components were held together with epoxy. The cell was then filled with the PMMA-dye solution. This approach failed, because of the formation of air bubbles after the evaporation of the solvent.

It was also found during this experiment that, due to the relatively low vapor pressure of toluene, the evaporation process was very slow. To save time in casting a film, methylene chloride was used to dissolve both 106-5 dye and PMMA in higher concentration. The solution was prepared by dissolving 9.4 milligrams of the dye in 90 ml of methylene chloride in the 100 ml glass stoppered flask. Twelve grams of PMMA was then added to the dye solution. A magnetic stirrer was then used to speed up the dissolving of PMMA. The solution was filtered through filter paper to remove any insoluble particles. A 5 mil thick film made from this solution was found to have a transmission of about 35 percent at 1.06 μm.

An improved technique for casting a good optical quality plastic film between two windows was to cast the film on a slide and polish its second surface until the film was about 5 mil thick. A second slide was then attached to form a sandwich as shown in Figure 18. The detailed procedure on making thin film is as follows:

a. Cut a 5 mil thick H-film into the size of a 1 inch x 3 inch slide, remove its center portion, roughly 1/2 inch x 2-1/4 inches.
b. Epoxy this rectangular H-film ring on one side of a clean slide.
c. Lay this slide flat in an evaporation dish with the H-film side up. Fill the area not covered by H-film with approximately 1 ml of the PMMA-dye solution, and then loosely cover the dish, so that the evaporation process will not be too fast. (Otherwise, evaporation will cool the material rapidly, and

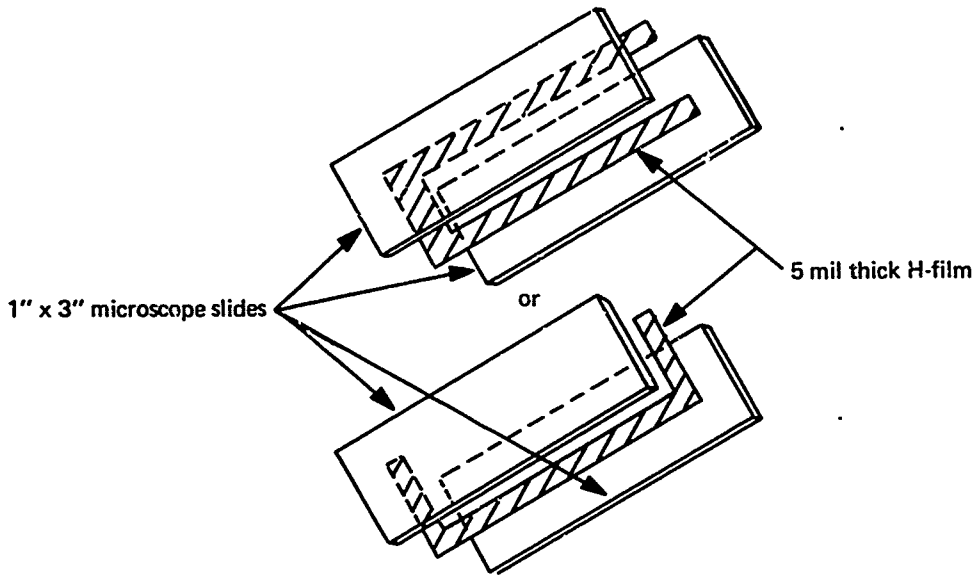


Figure 17. Construction of Thin Cell for Making Plastic-Dye Film AO0322-2

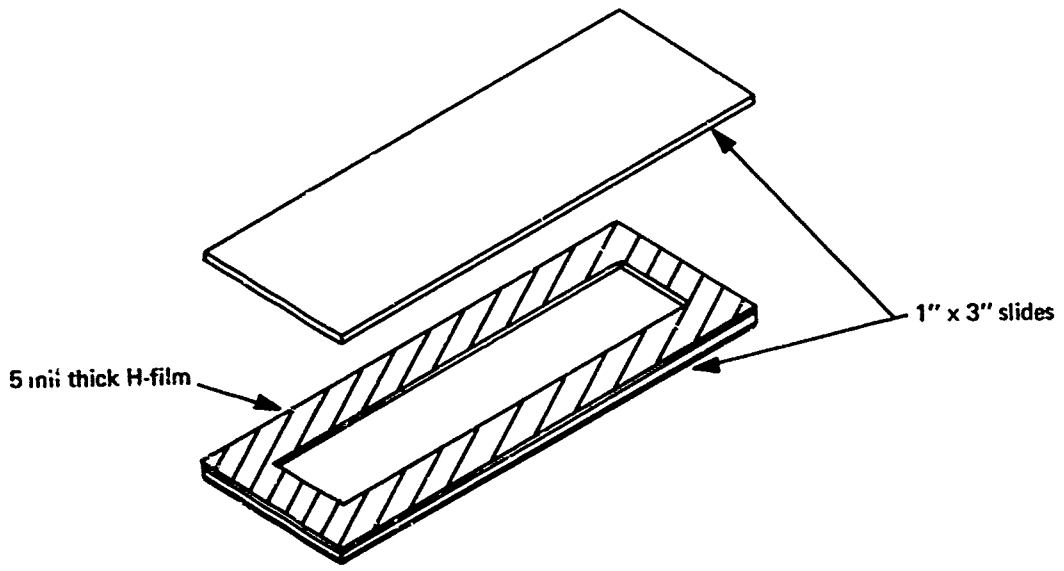


Figure 18. Construction of Supported Plastic-Dye Film AP0322-2

moisture will condense in the plastic compound, causing frost in the film.) Add more solution on it at least twice before the film gets hard (roughly once every hour) until the plastic material is thicker than 5 mils.

d. Polish the unsupported side of the film with a fine emery cloth, until it is slightly thicker than 5 mils, and then change to fine polishing compound (Edmund Scientific No. 401587), until it is as smooth as the surface of a glass slide.

4.2.3 Selection of Index Matching Materials

For polished films, the remaining scratches on the polished surfaces can be obviated as a source of loss by a supplemental trick of adding index matching material to fill up the air gap between the film and the slides. Several materials were tested for this purpose. They are described as follows:

a. Dip the polished film in methylmethacrylate monomer and polymerize the monomer in a slightly thicker cell made from two slides. This should give a perfect index match and also should not have the bubble forming problem, because there is no solvent to be evaporated during the polymerization. However, the result was unsuccessful, because the polymer film dissolved rapidly in the monomer.

b. Use clean epoxy (Emerson & Cuming Stycast 1269-A) to hold the film together with the two slides. This also failed, because curing of this epoxy required long time baking at 75°C (167°F), and the plastic material softened at that temperature and did not attach to the slides bubble-free.

c. Use Cargille immersion oil (Fisher Scientific No. 12-368-A) for index matching the film and glass. This unit was tested in a flashlamp pumped Nd:YAG laser and demonstrated its proper Q-switching property. However, the film started to show cracking after about half-an-hour. It probably indicates PMMA is slightly soluble in this oil.

d. Use ethylene glycol for index matching the film and glass. This unit also demonstrated its proper Q-switching property. The only disadvantage is the problem of leaking ethylene glycol.

e. Use room temperature curing clear epoxy (Emerson & Cuming Inc., ECCOBOND 24) to glue the film together with the slides. This film was tested in the flashlamp pumped Nd:YAG laser and worked well, except during the life test some minor damage was observed after over 500 shots at the power level of about 2 megawatts. This damage was probably due to the slight absorption of epoxy (less than 1 percent) at 1.06 μm.

f. Use Lens Bond (Summers Laboratories, Inc., Type F-65) to glue the film together with the slides. This film looked as good as described in the last paragraph but did not last for more than 20 shots in the test. Some cracks were found later in areas not used in this test. It was found later that this lens bond was a synthetic polyester adhesive; therefore, we suspected that it might have dissolved the PMMA-dye film before the polyester material was cured, just as happened in the case of polymerizing the monomer with the PMMA-dye film in it.

4.2.4 Heat Pressing Thick Sheet PMMA-Dye into Thin Film

To avoid using index matching material for improving the surface condition of the films, attempts were made to heat press a thick cast plastic sheet into thin film. The result was very encouraging, because it eliminated the tedious polishing process and resulted in smoother surfaces.

To cast a film of 0.5 mm thick with the solution containing 12 grams of PMMA in 90 ml of methylene chloride required filling the covered evaporation dish just one time to about 5 mm deep. This also avoided the moisture condensation introduced during the frequent filling made in the previous case. Solvent evaporation process took about two days. However, it is better to leave this cast plastic in a warm place for a few hours to drive the residual solvent out of the plastic before the start of heat pressing.

The technique of pressing involved just putting a small piece of the plastic between two clean microscope slides using a piece of 5 mil thick H-film as spacer. The assembly was secured with a spring loaded clamp and heated on a hot plate slowly to about 300 - 360 degrees F, until the plastic sheet was pressed to the thickness of the H-film. It was then cooled to room temperature and removed from the slides.

Films made in this way can be mounted on a metal ring holder. If liquid cooling is used for the laser, flowing coolant between laser rod, the Q-switch film, and the output mirror will not only furnish enough cooling to the film to make it suitable for multiple pulse purpose but also will make the lineup of the film less critical to the laser operation.

Section 5. OPERATIONAL TEST OF THE Q-SWITCH

5.1 106-5 DYE IN SOLUTION FORM

5.1.1 Test With a Nd:Glass Laser

The 106-5 dye dissolved in toluene was tested in a Nd:Glass laser. The same arrangement as shown in Figure 3 was used in the test, except that the resonator length in these tests was approximately 50 cm, the window of the Q-switch cell was slightly damaged due to previous tests, and two output mirrors with different transmissions were used. Some typical results are:

a.	Transmission of dye solution at 1.06 μm	70%
	Output mirror reflectivity (Salphire flat)	30%
	Output energy	0.5 joule
	Pulse duration	25×10^{-9}
	Slight modulation of output pulse observed	
b.	Transmission of dye solution at 1.06 μm	50%
	Output mirror reflectivity (dielectric coating)	45%
	Output energy	0.5 joule
	Pulse duration	10×10^{-9} sec.
	Slight modulation observed	

Typical signals are shown in Figure 19. Much lower output energy as compared to our 1969 result was mainly due to the extra scattering loss incurred at the damaged window of the dye cell. To prove this statement, 106-5 in 1,2-dichloroethane was used to replace the toluene solution. The same results were obtained.

5.1.2 Test with a Flashlamp Pumped Nd:YAG Laser

Q-switch solutions made with 1,2-dichloroethane and toluene as solvent, respectively, were both tested in a flashlamp pumped Nd:YAG laser for a 10 pulse per second experiment. This laser had a 1/4 inch x 3 inch rod with one of its end directly in contact with the dye solution. The maximum reflective mirror formed the other wall of the dye cell. Its output end was a 40 percent transmissive mirror. The Q-switch solution was circulated through the dye cell to avoid loss due to thermal distortion in the liquid. The input electrical energy was about 35 joules per pulse. Typical laser output energy was about 25 millijoules with a pulsewidth

less than 15 nanoseconds, when the unit was at single shot operation. No change in the beam pattern on an exposed Polaroid film was observed when the laser was switched to 10 pulse per second operation.

Further tests were performed on the toluene solution with this laser operated at 5 pulses per second for over 400,000 shots. No deterioration was observed, judging from the shape of the output pulses.

5.2 106-5 DYE IN SOLID FORM

5.2.1 Test With a Nd:Glass Laser

Two films of ∿0.1 mm thick, with a transmission of 74 percent at 1.06 μm, were cast in a dielectric film coated mirror. The plastic film did not adhere to the mirror coating well. Test was made on the same Nd: glass laser, except that the Q-switch dye cell was replaced with this plastic film coated mirror. The output mirror had 55 percent transmission at laser wavelength. With an electrical input energy of 3,000 joules, the laser output was greater than 1 joule. Due to the relatively high transmission for the films, the laser output consisted of multiple pulses. Modulation at the mode separation frequency appeared in most pulses, as shown in Figure 20. Damage was identified on these films after about 5 shots. The damage appeared in the form of wrinkling and microscopic pits in both surfaces of the films.

A thicker film (∿0.2mm) with a transmission of 42 percent at 1.06 μm was cast on a gold coated stainless mirror. This film was not bonded well to the mirror surface either. At the same input energy level, the Q-switched laser output was 0.85 joules and appeared as a single pulse of less than 10 nanoseconds in half-width. Damage to the film and mirror was noted after this single shot. The film cracked, and the mirror was severely burned, as shown in Figure 21. A second shot, using an undamaged portion of the mirror and with the film detached from the mirror surface, gave the same output energy. In this instance, the damage to the film was slight, but the mirror was again badly deteriorated.

A 5 mil thick film with ∿50 percent transmission at 1.06 μm was sandwiched between two microscope slides, index matched with clear epoxy, and tested in this laser. This film was inserted between the laser rod and the maximum reflection mirror. After a single shot, pits appeared in the film, which shows that this plastic Q-switch cannot be used to Q-switch a Nd:glass laser. The power density inside the resonator of this laser is estimated to be greater than 120MW/cm^2.

5.2.2 Test With a Flashlamp Pumped Nd:YAG Laser

A flashlamp pumped Nd:YAG laser with a 1/4 inch x 3 inch rod was used in this test. The output mirror was a 40 percent transmissive mirror. The input energy per pulse was 35 joules.

When a 0.2 mm thick film cast on a maximum reflective dielectric mirror was used, laser output consisted of multiple pulses. There was no visible damage to the film or mirror.

A similar 0.2 mm thick film was cast on a gold coated copper mirror. Multiple pulses were observed of less than 20 nanoseconds half-amplitude with an estimated peak power of a few megawatts. No damage to the plastic film was observed after 12 shots, but the gold mirror coating had a damaged spot of about 10 μm diameter.

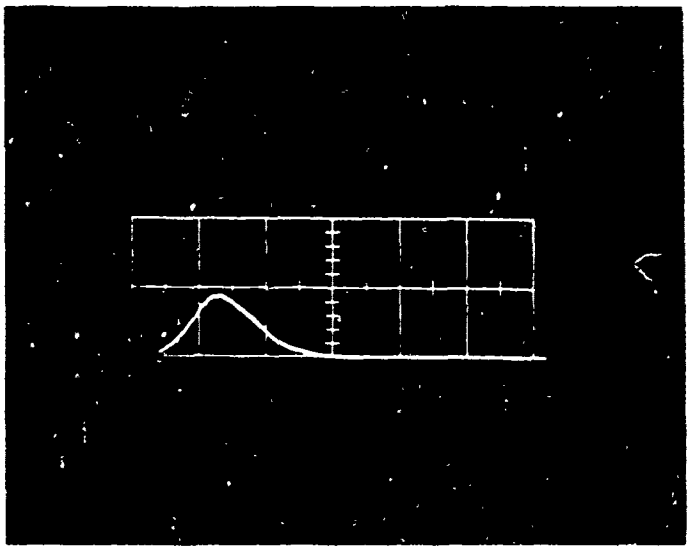
A 5 mil thick film with approximately 30 percent transmission at 1.06 μm was sandwiched between two pieces of microscope slides and index matched with clear epoxy (ECCOBOND 24). This film was tested in the Nd:YAG laser for more than 12 shots. Typical results showed single pulse output with pulse length of less than 15 nanoseconds and total energy of over 25 millijoules, which corresponded to a peak power greater than 2 megawatts. A picture of the Q-switched pulse, detected by a TRG 105B photodetector and displayed on a Tektronix 519 oscilloscope is shown in Figure 22a. The test arrangement is shown in Figure 22b. The film was checked under microscope (25x), and no visible damage was observed at the spot where the laser beam passed through, even after a dozen shots.

This film was also tested in the Nd:YAG laser at positions near the maximum reflective mirror, as well as near the output mirror. Comparing the burns on a piece of exposed Polaroid film, the laser worked better with the Q-switch film located near the output mirror, because it had a smaller beam, was more intense and had more uniform energy distribution.

A similar film sandwiched between two pieces of anti-reflection coated slides was also tested in this laser. The result was not very satisfactory because the AR coating on both slides was damaged after a few shots. It was found later that the coating on these slides was not suitable for high power application.

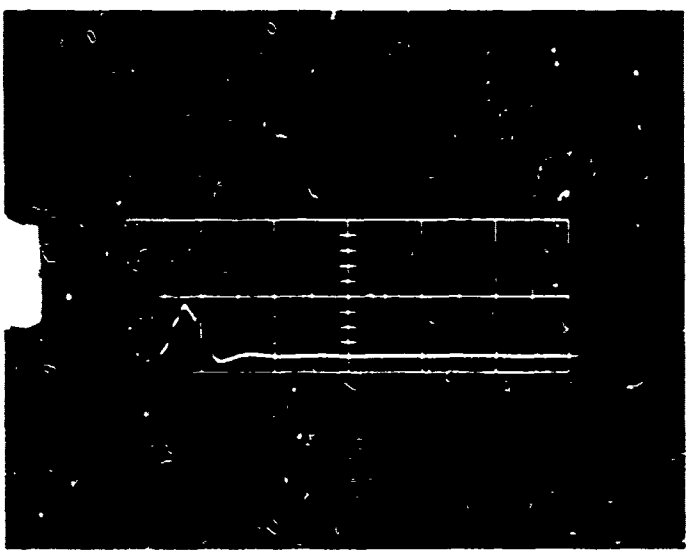
5.2.3 Temperature Effect and Life Test

Test on the temperature stability of this PMMA-dye film was done in two phases. For the temperature ranging from +20 degrees F (-7 degrees C) to +140 degrees F (+60 degrees C) the test was operational. A supported PMMA-dye film was used to Q-switch a flashlamp pumped Nd:YAG laser. The whole laser was installed inside a temperature controlled Tenney chamber. This



(d)

time: 20 ns/div; energy: 0.5 joule



(b)

time: 20 ns/div; energy: 0.45 joule

Figure 19. Output Pulses of the Nd:Glass Laser Using 106-5 Dye in Toluene as Q-Switch

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Time: 20 ns/div; Energy: 1.12 Joule

Figure 20. Output Pulses of the Nd:Glass Laser Using PMMA-Dye Film as Q-Switch

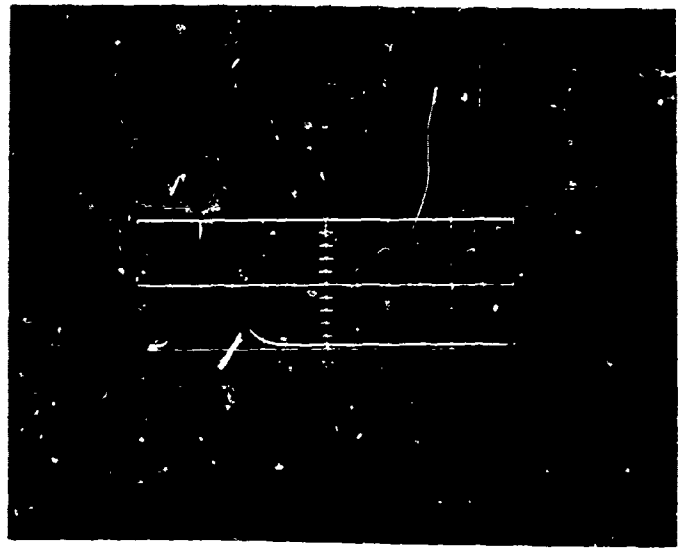
(a)

Damaged Postion of the PMMA-Dye Film

(b)

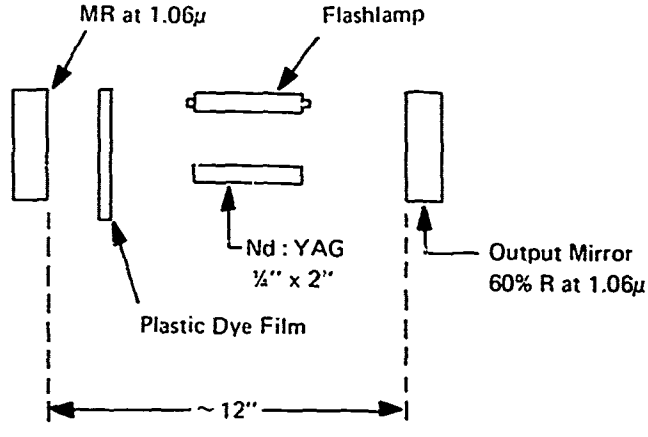
Damaged Portion of the Gold Coated Stainless Steel Mirror

Figure 21. Damage to the Plastic Film and Gold-Coated Mirror Due to the Q-Switched Pulse from an Nd:Glass Laser



(a) Typical Q-Switched Output

time: 20 ns/div. power: 2 megawatts
(energy: 25 millijoules)



(b) Test Arrangement

Figure 22. Output Pulse and Experimental Arrangement for Testing PMMA-Dye Film in Q-Switching a Flashlamp Pumped Nd: YAG Laser

laser consisted of a 2 inch x 1/4 inch rod, a 4 meter radius of curvature mirror with maximum reflection, and a flat mirror with 37 percent transmission at 1.06 μm. The Q-switch film was located between the output mirror and the laser rod. The experimental arrangement is shown in Figure 23. Laser output was coupled out through the chamber window via three rectangular prisms. The signal was detected by a TRG 105B photodetector and displayed on a Tektronix 519 oscilloscope. The test started with the highest temperature, and the temperature was reduced at 20 degrees F (11 degrees C) steps. Measured by the photodiode response, the laser output power showed a consistent increase when the chamber temperature was reduced. This increase in output power at lower temperature was credited to the achievement of higher population inversion in the laser medium.

Test at temperatures below 20 degrees F was nonoperational. It was done by storing the supported film in a freezer at a temperature below -40 degrees F for about an hour and then warmed up to room temperature. No physical change was observed during this temperature change. This film was then used to Q-switch a laser operated at room temperature. No significant change in the Q-switched output was observed.

Life test on the supported PMMA-dye film was done with the same Nd:YAG laser. The laser cavity length was approximately 1 foot. Its output energy and pulse shape were detected by a TRG 100 energy meter and a TRG 105B photodetector, respectively. The output pulses had a total energy of 20 millijoules and a pulse length of less than 10 nanoseconds. A plot of laser output energy distribution for 609 events taken during this test is shown in Figure 24.

This Q-switch film was inspected microscopically before and after the test. After over 500 shots were made at the same spot, no visible damage could be seen by naked eye. No significant change in pulse shape and output energy were observed. However, under 100 times magnification, pits of about 5 μm in diameter (or smaller) were observed on both surfaces of the film. The damage on opposite surfaces did not have the same pattern as shown in Figure 25, and no damage between the two surfaces was observed. This suggests that the damage probably occurred in the epoxy adhesive layers.

To find out whether previously observed damage was in the epoxy layers or on the surfaces of the PMMA-dye films, further life test was done on unsupported films. About 120 shots were made on an unsupported film and no damage was observed after the test. This gave us reasonable indication that the damage problem was in the epoxy.

Unsupported films made through heat pressing process in the later stage were also tested. These films Q-switched the laser with more uniform burn on an exposed Polaroid film. A film of ~15 mils thickness made in this way was also tested for multiple pulse operation at up to three pulses per second. In this test, the PMMA-dye film was immersed in FC-

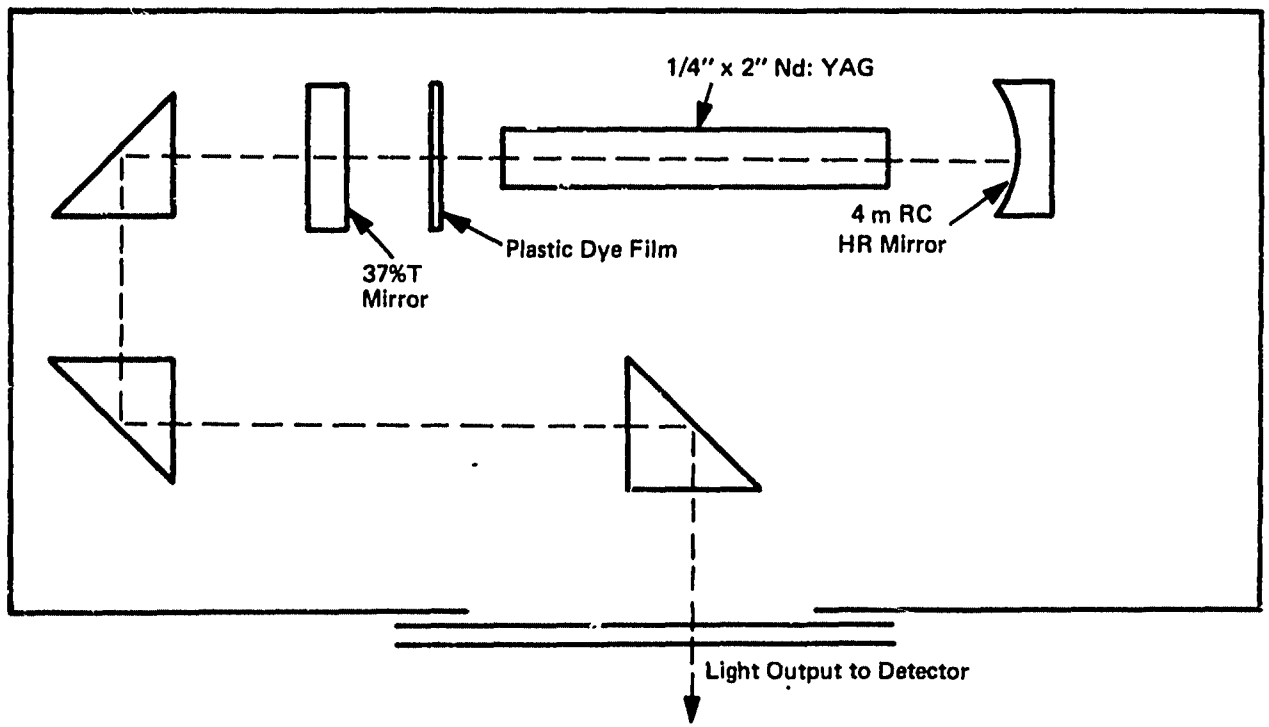


Figure 23. Experimental Arrangement for Testing the Temperature Effect on the Q-Switching Plastic-Dye Film

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104 (fluoro-carbon) coolant. The coolant was circulated through the system with a small pump. Since input energy per pulse used in this test was about 45 joules, which was way above the maximum allowable energy one could put into this particular laser, problems with the pump cavity developed. To reduce the input energy to within its safe operational level, films with lower dye concentration should be fabricated for further test in the future.

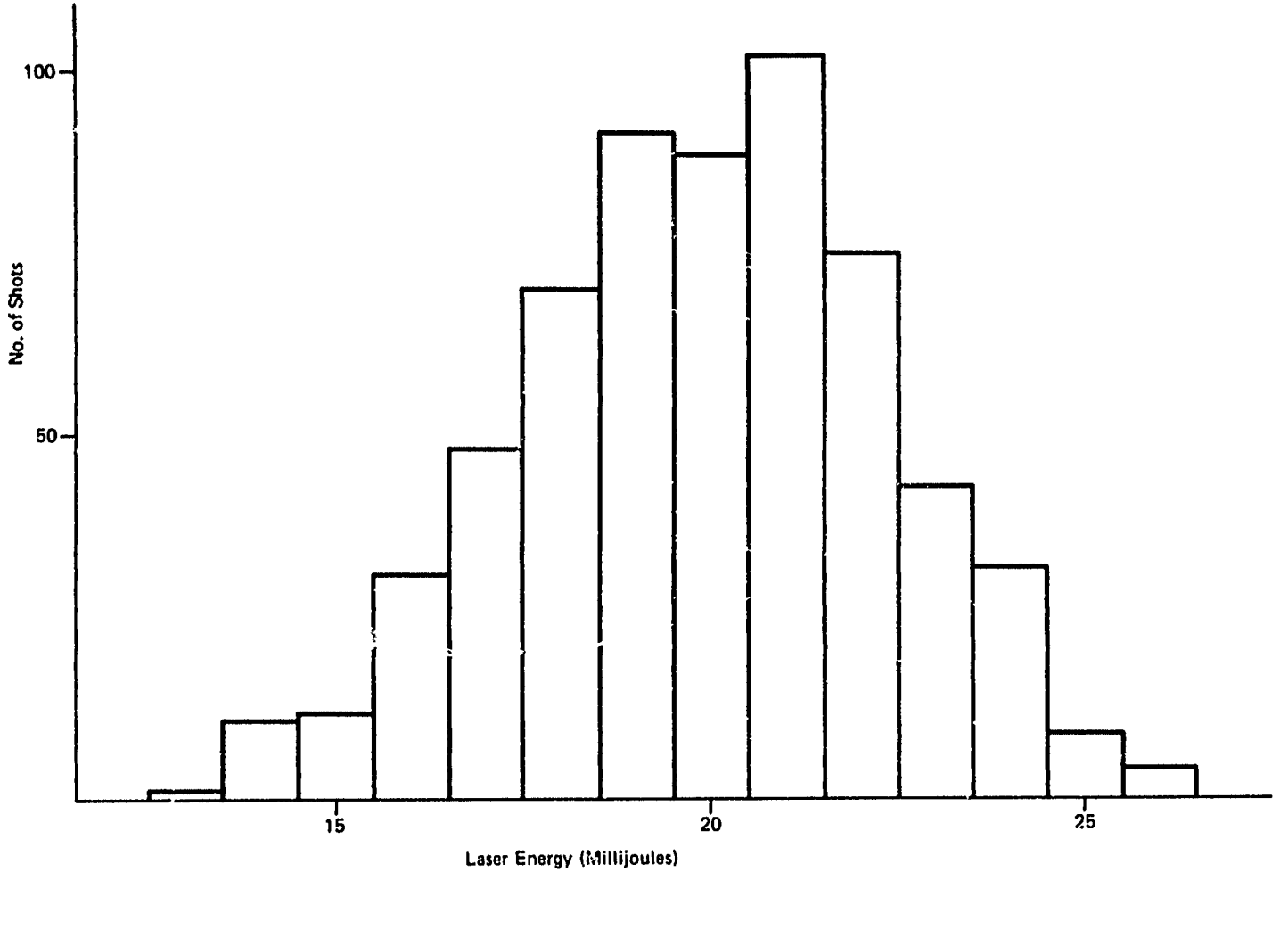

Figure 24. Result of Life Test on Using PMMA-Dye Film to Q-Switch a Flashlamp Pumped Nd:YAG Laser

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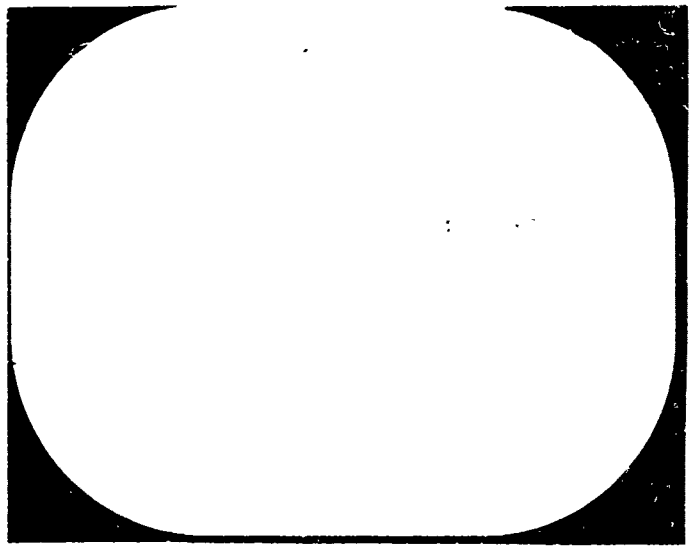

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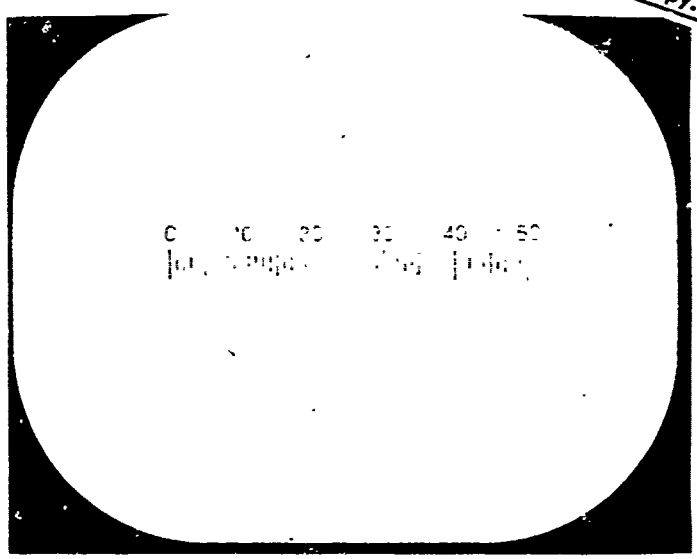

X 100

Figure 25. Damage to the Surfaces of the Supported Q-Switching Film

Section 6. CONCLUSIONS AND RECOMMENDATIONS

This program has shown clearly that the IBM 106-5 dye dissolved in 1, 2-dichloroethane or in toluene is an excellent passive Q-switch for neodymium lasers. In either solvent, high energy (or 2 joules/pulse) and high power (∿ 100 MW) Q-switching can be performed with no observable degradation of the material. Specifically, Life tests at 1 joule/cm^2 energy density have shown no degradation of performance after 1000 Q-switched pulses. Tests also showed that the Q-switch solution will not deteriorate under ambient radiation for at least a year. A temperature stability test has shown that for a properly sealed dye cell there is no need to readjust the dye concentration for obtaining repeatable Q-switched laser output throughout the MIL-STD-810B operational and storage temperature range. But provision should be made for varying the input power to the flashlamp to compensate the change in laser threshold due to the change in ambient temperature.

Of the two solvents mentioned in the last paragraph, toluene is preferred over 1, 2-dichloroethane because the 106-5 dye is more soluble in toluene which allows a greater range of concentrations to be used. Also, toluene provides wider operational temperature range and less toxicity. Furthermore, from the test results of epoxy adhesives, toluene will not attack either Hysol 931 or Hysol 956, so the dye cell can be made more cheaply.

Because of its high durability, the 106-5 dye solution can also be used to Q-switch neodymium lasers at a high pulse repetition rate. Satisfactory operation was achieved in Q-switching a flashlamp pumped Nd:YAG laser to over 20 pulses per second with an output energy of over 30 millijoules per pulse. The limitation to this experiment was not the dye solution but the power supply used to energize the flashlamp. Operation in the multiple pulse mode requires provision for circulating the dye solution through the Q-switch cell to avoid thermal distortion resulting from absorption.

An important result of this program is dispersion of the 106-5 dye into polymethylmethacrylate PMMA. The plastic Q-switch offers advantages over the liquid Q-switch in size and ease in handling.

Both supported and unsupported PMMA-dye films were used to Q-switch a flashlamp pumped Nd:YAG laser at about 2 MW output power. A supported film put together with ECCOBOND 24 epoxy adhesive and microscope slides was slightly damaged after 500 shots. This damage was identified later to be in the adhesive layers. An unsupported film tested over 120 shots under the same condition did not show any damage. Unsupported film with coolant flowing over both surfaces was tested briefly in Q-switching a multiple pulse Nd:YAG laser and proved to be feasible for that application, if the laser peak power is limited to less than 1 MW. A suitable

heat pressing process was found for making the plastic-dye films. However, the technique used in this process was primitive and not well controlled. Further improvements in this area can be expected, from expert plastic material handling to development of better pressing techniques.

Two specific types of continuation have been suggested by this work. Much effort in laser engineering has been directed toward design of a high-power, single-mode Q-switched laser. The passive Q-switch developed under the program can be utilized in such a design to produce better results than previously achieved. An area of research interest which holds promise for further advantages concerns passive mode-locking of C.W. neodymium lasers. Mode-locking of pulsed lasers has been observed with the 106-5 dye and recent results in passive mode-locking for C.W. lasers* suggest that analogous work is possible with Nd:YAG. Such an advance could be widely used in such applications as communications.

*C.V. Shank, E.P. Ippen, and A. Dienes, Passive Mode-locking of the CW Dye Laser, VII International Quantum Electronics Conference, 1972.